INTERLABORATORY STUDY 89-1

VOLATILE ORGANIC PARAMETERS IN REAGENT WATER AND OIL AND GREASE IN REAGENT WATER AND EFFLUENT

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VOLATILE ORGANIC PARAMETERS IN REAGENT WATER

AND OIL AND GREASE IN REAGENT WATER AND EFFLUENT

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1 SUMMARY AND CONCLUSIONS

Interlaboratory Study 89-1 was the second of an on-going program of laboratory performance management studies initiated by the Quality Assurance Office, Laboratory Services Branch of the Ontario Ministry of the Environment. It assesses the analytical variability of selected organic parameters (see Sections 3.1.2 and 3.1.3) in spiked reagent water and Oil and Grease (Solvent Extractables) from reagent water and one effluent sample. Sixteen laboratories initially agreed to participate in the study. Results for the Volatile Organic samples were reported from ten participants. Results for the Oil and Grease samples were reported from twelve of the participants.

The results demonstrate variable performance between laboratories. Some participants were able to achieve good recovery (80-110%) of the target parameters across the scan. Other laboratories demonstrated biases due to differences in calibration standards or analytical procedures.

Differences in standards may result in consistent over- or under-recovery across the scan. Differences in analytical procedures may produce a variety of effects, including over- or under-recovery of specific parameters, patterns of increasing or decreasing recovery across the scan, and variability of recovery of low level spikes versus high level spikes. It is desirable that laboratories demonstrating some of these problems strive to correct these difficulties. Future interlaboratory studies that include the analysis of Volatile Organics will attempt to monitor improvements in performance by the participants.

Due to the large number of parameters included in the Volatile scan, this report does not attempt to assess the between-laboratory performance of individual parameters. A review of the range of results given by the Minimum and Maximum values in Table 1, Appendix 1, indicates that the results may vary by an order of magnitude. However, as there were only two spiked samples submitted to each participant, there is insufficient data to assess the analytical characteristics of each parameter. Future studies will attempt to assess the analytical characteristics of individual parameters by submitting more samples to the participants and including duplicate analyses.

The results for the Oil and Grease samples demonstrate that, regardless of solvent used, individual laboratories demonstrate systematic bias. The participants demonstrated good within-laboratory precision, but the between-laboratory variability had a significant effect on the data distribution. The analytical techniques used to perform this test may affect the variability of performance (3) between the participants. Future studies will attempt to monitor improvements in performance for this analytical method.

The following table summarizes the individual laboratory's performance.

TABLE I - SUMMARY OF PARTICIPANTS' PERFORMANCE

LAB CODE	MISA TEST GROUP	PERFORMANCE
1001	Volatiles (16 & 17)	- Results from only one sample (second sample broken in transit), therefore not possible to assess within-laboratory precision
		- Variable performance across scan
		- Reported MDL's for Bromomethane, Chloromethane and Vinyl chloride greater than specified in MISA General Regulation, therefore not possible to determine if there was background contamination for these parameters
	Oil and Grease (25)	- Excellent recovery relative to design values but appear high relative to other participants
		- Possible background laboratory contamination
1002	Volatiles (16 & 17)	- Good recovery relative to design values
		- Consistent performance across scan except for 5 specific parameters
		- Some laboratory contamination with Dichloromethane, but at a very low level
	Oil and Grease (25)	- Excellent recovery relative to design values but appear high relative to other participants
		- No evidence of laboratory background contamination
1003	Volatiles (16 & 17)	- Good recovery relative to design values
		- Consistent performance across scan except for 5 specific parameters
		- No evidence of laboratory background contamination
	Oil and Grease (25)	- Variable performance: good recovery of the low spike but under-recovered the high spike
		- No evidence of laboratory background contamination

LAB CODE	MISA TEST GROUP	PERFORMANCE
1004	Volatiles (16 & 17)	- No results reported for these samples
	Oil and Grease (25)	- Good recovery relative to the design values
		- Good agreement with the interlaboratory mean and median
		- No evidence of laboratory background contamination
		- Very high result for the effluent sample; result was deleted from statistical calculations
1005	Volatiles (16 & 17)	- Variable within-laboratory precision
		- Good recovery of low spike for most parameters but many early-eluting parameters under-recovered in high spike
		- Consistent pattern of recovery across scan for low spike but pattern of increasing recovery across scan for high spike
		- No evidence of laboratory background contamination
	Oil and Grease (25)	- No results reported for these samples
1007	Volatiles (16 & 17)	- Biased low relative to design values and interlaboratory mean
		- Very low background level of Dichlorobenzes but no other evidence of background contamination
		- Pattern of increasing recovery across the scan in low spike but no pattern demonstrated in high spike
	Oil and Grease (25)	- Excellent recovery relative to design values but appear high relative to other participants
		- Good within-laboratory precision
		- No evidence of laboratory background contamination

LAB CODE	MISA TEST GROUP	PERFORMANCE
1008	Volatiles (16 & 17)	- No results reported for these samples
	Oil and Grease (25)	- Biased low relative to the design values and the interlaboratory mean - Good recovery relative to the interlaboratory median - Good within-laboratory precision - No evidence of laboratory background contamination
1009	Volatiles (16 & 17)	- Good recovery relative to the design values for the low spike but biased low for the high spike - Variable within-laboratory precision - Some laboratory contamination with Toluene, but at a very low level
	Oil and Grease (25)	- Good recovery of the low spike relative to the design value but under-recovered the high spike - Low spike result was in good agreement with the interlaboratory mean and median, but biased low for the high spike - No evidence of laboratory background contamination

LAB CODE	MISA TEST GROUP	PERFORMANCE
1010	Volatiles (16 & 17)	- Variable performance relative to the design values - Pattern of recovery across the scan similar for both spiked samples - Laboratory contamination with Dichloromethane
	Oil and Grease (25)	- Biased low relative to the design values and the interlaboratory mean and median - Consistent within-laboratory performance - Some possible laboratory background contamination
1011	Volatiles (16 & 17)	- Did not participate in this portion of the study
	Oil and Grease (25)	- Good recovery of the high spike relative to the design value but under-recovered the low spike - High spike result was in good agreement with the interlaboratory mean and median, but biased low for the low spike - No evidence of laboratory background contamination
1012	Volatiles (16 & 17)	- Variable recovery relative to the design values - Similar pattern of recovery across the scan demonstrated in both spiked samples - Unusual laboratory contamination with Chloromethane - Laboratory contamination with Dichloromethane
	Oil and Grease (25)	- No results reported for these samples

LAB CODE	MISA TEST GROUP	PERFORMANCE
1013	Volatiles (16 & 17)	- Good recovery relative to design values - Consistent performance across scan except for 3 specific parameters - No evidence of laboratory background contamination
	Oil and Grease (25)	- Biased low relative to the design values and to the inter-laboratory mean and median - Variable within-laboratory precision - No evidence of laboratory background contamination
1014	Volatiles (16 & 17)	- Variable recovery relative to the design values - Laboratory contamination with Dichloromethane - Recovery across scan clusters into three groups; demonstrated in both spiked samples
	Oil and Grease (25)	- Biased low relative to the design values - Good recovery relative to the interlaboratory mean and median - Good within-laboratory precision - No evidence of laboratory background contamination
1015	Volatiles (16 & 17)	- No results reported for these samples
	Oil and Grease (25)	- Good recovery relative to the design values - Good recovery relative to the interlaboratory mean and median - Good within-laboratory precision - No evidence of laboratory background contamination

2 INTRODUCTION

Interlaboratory performance studies, or round robins, are conducted to assess the comparability of data among different laboratories. These studies help in the identification of biases and precision or accuracy problems. Participation in such studies helps in improving individual laboratory performance and maintaining performance standards. The Quality Assurance Office, Laboratory Services Branch (LSB) of Environment Ontario has instituted an on-going program of interlaboratory studies to assess and enhance the performance of environmental laboratories providing analytical services.

Interlaboratory Study 89-1 consists of two components. Part 1 was designed to assess the analytical variability of volatile organic parameters in fortified reagent water. Part 2 was designed to assess the analytical variability of Oil and Grease (solvent extractables) from fortified reagent water and an effluent sample. The parameter list was chosen from analytical test groups listed in the MISA (Municipal and Industrial Strategy for Abatement) General Regulation (1). Participants were requested to use methods which conformed to the MISA analytical principles and protocols given in the General Regulation (1).

Sixteen laboratories agreed to participate in this interlaboratory study. A list of participants is included in Appendix 2. Not all of the participants produced final results for all of the samples submitted (see Section 3.4). Final percent participation was as follows: Volatiles - 69%, Oil and Grease - 75%.

Two sets of samples (7 in total) were distributed to each of the 16 participants. Each set consisted of three samples for volatile organic analysis (MISA Test Groups 16 and 17), and four samples for Oil and Grease analysis (MISA Test Group 25). Details of sample preparation and distribution are given in Sections 3.1 and 3.2. Analytical methodology and data handling are presented in Sections 3.3 and 3.4. Final results are presented and laboratory performance is discussed in Section 4.0.

3 PROCEDURE

3.1 Preparation of Samples

3.1.1 Preparation of Matrices

a) Reagent Water

Volatile Samples

Four litres of deionized, distilled water was collected in a clean Erlenmeyer flask. It was purged with a gentle stream of nitrogen gas for approximately two hours. This process was carried out in a separate laboratory, well removed from routine organic laboratory atmosphere to avoid contributing artifacts to the analysis. Any contact of water with plastic materials was avoided.

Oil and Grease Samples

Non-purged, deionized, distilled water was used for the Oil and Grease samples. Care was taken that no plastic materials came into contact with the water.

b) Effluent Matrix

A bulk effluent sample was collected from extra samples received at Laboratory Services Branch. The effluent was stirred overnight to form a homogeneous mixture using a stirrer with a Teflon shaft and propeller.

3.1.2 Preparation of Volatile Samples

A combined spiking solution was prepared in purged, distilled, deionized water. Stock solutions consisted of three different volatile organic mixtures purchased from Supelco, and one in-house prepared ampoule. Table II lists the mixtures of organic parameters stated to be present in the ampoules by the supplier.

TABLE II - VOLATILE ORGANICS PARAMETER LIST

Purgeable A	Carbon Tetrachloride
	Chlorobenzene
	2-Chlorovinyl ether
	Chloroform
	Dibromochloromethane
	1,1,-Dichloroethane
	1,1-Dichloroethylene
	1,2-Dichloropropane
	Methylene chloride (Dichloromethane)
	Tetrachloroethylene
	1,1,2-Trichloroethane
	Trichloroethylene
	Trichlorofluoromethane
Purgeable B	Benzene
	Bromodichloromethane
	Bromoform
	1,2-Dichloroethane
	t-1,2-Dichloroethylene
	1,3-Dichloropropene (both isomers)
	Ethyl benzene
	1,1,2,2-Tetrachloroethane
	Toluene
	1,1,1-Trichloroethane
Purgeable C	Bromomethane
	Chloroethane
	Chloromethane
	Vinyl chloride
VOLA02 (In-house ampoule)	p-Xylene
	m-Xylene
	o-Xylene
	1,2-Dichlorobenzene
	1,3-Dichlorobenzene
	1,4-Dichlorobenzene
	Ethylene dibromide

Samples for volatile analysis were prepared in 40 mL glass vials that had screw caps with Teflon-lined septa. Sample vials were filled almost to overflowing with the sample matrix, with the miniscus rising above the edge of the vial. The volume of 40 mL was confirmed by weighing the vials before and after filling (±1 mL). Unspiked samples were labelled "VOL A".

All sample spiking was done using 50 or 500 microlitre syringes, depending on the spiking level. The spiking solution was expelled from the syringe below the surface of the matrix. The screw cap septa (Teflon side towards the sample) was carefully slid across the top of the

vial and quickly screwed on, so as to avoid trapping any air bubbles in the vial. The samples were gently inverted three or four times to mix the contents. The low spike samples were labelled "VOL B" and the high spike samples were labelled "VOL C".

3.1.3 Preparation of Oil and Grease Samples

Samples for Oil and Grease analysis were prepared using amber 1000 mL glass bottles. To each sample bottle, 1000 mL of distilled, deionized water was added by weight (1000 g ±2 g). It was assumed that the density of the water equalled 1.0. The unspiked samples were labelled "O&G A".

A spiking solution was prepared by weighing an appropriate amount of cooking oil in a volumetric flask, and diluting to volume with acetone. Spiking was done using 0.5 and 1.0 mL gas-tight syringes. The low spike was labelled "O&G B" and the high spike was labelled "O&G C".

The homogenized bulk effluent was added to each sample bottle by weight (1000 g ±2 g). Any possible error introduced by the density of the effluent was considered too small to be significant. These samples were not spiked with the solution used for the reagent water samples. They were labelled as "O&G D".

3.2 Sample Distribution

Prior to sample preparation a letter of notification was sent to each participating laboratory. Confirmation of participation was received by letter or telephone. A list of participating laboratories and examples of correspondence is given in Appendix 2.

The two sets of three and four samples (Volatiles and Oil and Grease respectively) for each participating laboratory were packaged in cardboard boxes and shipped via Purolator courier on January 31, 1989. One laboratory reported that one cap came off one sample bottle for Oil and Grease during transit. Another laboratory reported that one sample for Volatiles was broken in transit. A third laboratory reported that they did not receive the package containing the Volatile samples. These samples were not replaced.

3.3 Analytical Methodology

Participants were requested to analyze the samples using routine in-house methods that complied with the principles and protocols outlined in Schedule 3 of the MISA General Regulation (1).

Participants were not required to provide detailed information regarding instrumental methodology. Some participants provided information regarding instrument model and column used, and all participants stated that additional information was available on request. Participants were

asked to indicate which solvent was used for the Oil and Grease analyses. Participants were subsequently contacted and requested to indicated which type of gas chromatograph column had been used for the analysis of the Volatile Organics.

3.4 Data Handling

Results were submitted to the Quality Assurance Office, LSB in written form by mail. All data were manually entered by laboratory code into an electronic spreadsheet. Blank spaces were left when a laboratory did not report results for a specific parameter that was present in the spiking material. A "0" was entered when a laboratory reported values for a specific parameter in some samples, but reported "Not Detected" in other spiked samples.

Final percent participation was as follows: Volatiles - 69%, Oil and Grease - 75%.

Interlaboratory variability was determined by calculating the mean, median, and standard deviation from the results reported (n=11 for Volatiles). The minimum and maximum values were identified to give an indication of the range. Outliers were not removed from the data set when calculating interlaboratory variability as the range of results precluded a selection of participants demonstrating a central tendency.

The results for the Oil and Grease were divided into two groups, depending on the solvent used for the extraction by the participants. The mean, median, minimum, maximum, and standard deviations were calculated for each group (n=7 for Freon and n=5 for Dichloromethane). Sample O&G A (Blank Reagent Water), had several results reported "< value". To perform the calculations, one half of the reported "value" was used. One result for O&G D was deleted when calculating the mean and standard deviation from the participants using Dichloromethane.

Results were converted to percent recovery based on the design value of the spiking material for both data sets. These values are presented in Tables 2 and 4 in Appendix 1. The results were not blank corrected.

Appendix 1 includes bar graphs of the recovery for each parameter from each participating laboratory (Figures 1-6 for Volatiles and Figures 7-8 for Oil and Grease). For the Volatiles, parameters are arranged left to right in order of gas chromatographic elution (based on a SPB-5 capillary column). Outliers were not deleted from the data set when preparing the graphs. All results are presented as percent recovery relative to the design value. Different participants reported the results for o-, m-, and p-Xylenes in varying combinations. The graphs have been separated as follows:

(i) Figure 1 has two laboratories reporting the three isomers separately,

(ii) Figures 2, 3, 5, and 6 have seven laboratories reporting m- and p-Xylenes together and o-Xylene separately, and

(iii) Figure 4 has one participant reporting o- and p-Xylenes together and m-Xylene separately.

Figure 9 is a bar graph of the results from each participant for the bulk effluent sample (O&G D). Results are presented as mg/L and separated according to the two different solvents used.

4 RESULTS AND DISCUSSION

An overview of the interlaboratory performance for Volatiles is described. Individual performance of each participant is described separately and was assessed according to the following criteria: recovery of each parameter relative to the design values, laboratory background contamination of selected parameters, and consistency of performance within the scan (based on a SPB-5 capillary column).

On overview of the interlaboratory performance for Oil and Grease is described. Individual performance of each participant is described separately and was assessed according to the following criteria: recovery relative to the design values and performance relative to the interlaboratory mean and median.

4.1 Volatile Samples

OVERVIEW OF INTERLABORATORY PERFORMANCE - VOLATILES

The results demonstrate variable performance between laboratories. Some participants were able to achieve good recovery (80-110%) of the target parameters across the scan. Other laboratories demonstrated biases due to differences in calibration standards or analytical procedures.

Differences in standards may result in consistent over- or under-recovery across the scan. Differences in analytical procedures may produce a variety of effects, including over- or under-recovery of specific parameters, patterns of increasing or decreasing recovery across the scan, and variability of recovery of low level spikes versus high level spikes. It is desirable that laboratories demonstrating some of these problems strive to correct these difficulties. Future interlaboratory studies that include the analysis of Volatile Organics will attempt to monitor improvements in performance by the participants.

Due to the large number of parameters included in the Volatile scan, this report does not attempt to assess the between-laboratory performance of individual parameters. A review of the range of results given by the Minimum and Maximum values in Table 1, Appendix 1, indicates that the results may vary by an order of magnitude. However, as there were only two spiked samples submitted to each participant, there is insufficient data to assess the analytical characteristics of each parameter. Future studies will attempt to assess the analytical characteristics of individual parameters by submitting more samples to the participants and including duplicate analyses.

LABORATORY 1001

Sample VOL C (High Spike) was broken in transit and therefore results were not reported for this sample. This laboratory did not report results for Trichlorofluoromethane. They used a DB-1 60 m, 0.25 mm I.D. capillary gas chromatographic column.

Recovery of the parameters from Sample VOL B was variable, relative to the design values. Early eluting parameters were over-recovered, except for 1,1-Dichloroethylene. Late eluting parameters were under-recovered, particularly trans-1,3-Dichloropropylene, Tetrachloroethylene, Chlorobenzene, and 1,1,2,2-Tetrachloroethane.

Results for the blank sample (VOL A) were all reported as <MDL. For most parameters, the results suggest that there was no background contamination in this laboratory. For three parameters (Bromomethane, Chloromethane, and Vinyl chloride) the MDL was <10 µg/L. The MISA Regulation MDLs are 3.7, 3.7, and 4.0 respectively. It is difficult to determine whether there was any background contamination for these three parameters.

Performance across the scan was very variable. As results were reported from only one sample (VOL C was broken in transit), it is not possible to assess the consistency of performance of this laboratory.

LABORATORY 1002

This laboratory did not report any problems with the analysis of the samples. They used a DB-5 capillary gas chromatographic column.

The results demonstrated good recovery relative to the design values, for the majority of the parameters. Recovery varied between 85-115% for both the low and high spiked samples (VOL B and VOL C, respectively). There were some problem parameters that were under-recovered, particularly Trichlorofluoromethane, Carbon Tetrachloride, Tetrachloroethylene, and m- & p-Xylenes. Chloromethane and 1,2-Dichlorobenzene were over-recovered by a noticeable amount.

A small amount (1.7 µg/L) of Dichloromethane was reported in the blank sample (VOL A). This is a common laboratory contaminant, but does not appear to pose a problem. The results for Dichloromethane in the spiked samples were not corrected by this value.

Some parameters appear to present some problems for analysis by this participant. As mentioned above, there were several parameters that were under-recovered (<60% recovery). The patterns of recovery were the same in both the low and high spiked samples (Figures 2 and 5). These results suggest that this laboratory has consistent performance, but needs to address some specific problem parameters.

LABORATORY 1003

This laboratory did not report any problems with the analysis of the samples. They used a J & W DB-624 30 m, 0.032 I.D. capillary gas chromatographic column.

The results demonstrated good recovery relative to the design values, for many of the parameters. Recovery ranged between 80-110% for both the low and high spiked samples (VOL B and VOL C, respectively). 1,1,2-Trichloroethane and Ethylene Dibromide were over-recovered in both samples (140% and 136% in the low spike, and 168% and 149% in the high spike). Low recovery (<60%) was observed for Trichlorofluoromethane, 1,1-Dichloroethylene, and Carbon Tetrachloride in both samples.

Nothing was reported in the blank sample (VOL A), so there was no evidence of any laboratory contamination problems.

Similar patterns of recovery were observed for both the low and high spiked samples (Figures 2 and 5). The same parameters were over- or under-recovered in both samples, as noted above. These results suggest that this laboratory has consistent performance but needs to address some specific problem parameters, as noted above.

LABORATORY 1005

This laboratory did not report any problems with the analysis of the samples. They used a SE-30 30 m, 0.025 mm I.D. capillary gas chromatographic column.

The results for the low spike (Sample VOL B) demonstrate good recovery relative to the design values, for most of the parameters. Several parameters were under-recovered, particularly Trichlorofluoromethane, 1,1,-Dichloroethylene, Carbon Tetrachloride, and Tetrachloroethylene. The recovery (relative to the design values) was not consistent in the high spike (VOL C). Many parameters, particularly at the beginning of the scan, were under-recovered.

Nothing was reported in the blank sample (VOL A), so there is no evidence of any laboratory contamination problems.

The results across the scan for the low spike present a consistent pattern of recovery (Figure 2), except for the problem parameters noted above. However, for the high spike there is a pattern of increasing recovery (Figure 5), except for Tetrachloroethylene. These results suggest that this laboratory has some difficulty analyzing samples of a higher concentration.

LABORATORY 1007

This laboratory did not report any problems with the analysis of these samples. They used a DB-5 30 m capillary gas chromatographic column.

The results from both spiked samples were under-recovered relative to the design values, except for o-Xylene in the low spike (VOL B). These results were also low relative to the interlaboratory mean for both samples. This suggests that their calibration standard differs from the spiking material used by the QA Office of MOE and also differs from the calibration standards of the other participants.

Values of less than 1.0 µg/L were reported for the three Dichlorobenzenes in the blank sample (VOL A). Values this low should not present any problems of laboratory contamination. No other evidence of laboratory contamination is apparent in the results.

The results from the low spike demonstrate a pattern of increasing recovery across the scan (Figure 3). The loss of the first seven parameters in the scan from this sample suggests that some adjustments are necessary to the temperature programming. This pattern is not evident in the results from the high spike (Sample VOL C, Figure 6), but there are several early eluting parameters that had very low recoveries (<20%). Some individual parameters in the middle of the scan also appear to be difficult for this laboratory to analyze, particularly Carbon Tetrachloride and trans-1,3-Dichloropropylene. There are several problem areas that need to be addressed by this laboratory.

LABORATORY 1009

This laboratory did not report any problems with the analysis of these samples. They used a Restek TRX-Volatiles 60 m, 0.032 mm I.D. capillary gas chromatographic column.

The results for the low spike (Sample VOL B) demonstrate good recovery relative to the design values, for most of the parameters. Four parameters were under-recovered (Trichlorofluoromethane, Dichloromethane, Carbon Tetrachloride, and Tetrachloroethylene). Bromomethane was over-recovered in both spiked samples. The results from the high spike (VOL C) were low, relative to the design value. The same parameters that had low recoveries for the low spike were also much lower for the high spike.

A value of 1.1 µg/L was reported for Toluene in the blank sample (VOL A). This is a common laboratory contaminant, but at this low level does not appear to pose a problem. The value reported in the blank for Toluene was not subtracted from the results for Toluene in the spiked samples.

The results across the scan for the low spike present a consistent pattern of recovery (Figure 3), except for the problem parameters noted above. The results across the scan for high spike are more variable (Figure 6). This suggests that this laboratory has more difficulty analyzing samples of a higher concentration. This may be due to curvature at the top end of their calibration range.

LABORATORY 1010

Results were not reported for the following parameters present in the spiking material: 1,2-Dichloroethane, Bromomethane, Chloromethane, trans-1,3-Dichloropropylene, and Vinyl Chloride. They used a 1% SP-1000 Carbopak gas chromatographic packed column. Schedule 3 of the MISA General Regulation (1) specifies the use of a capillary column. For regulatory purposes, this laboratory's results would not be acceptable. The results are reviewed for the purposes of improving laboratory performance.

The results for the low spike (VOL B) were under-recovered relative to the design value, for most parameters, except for Dichloromethane, cis-1,3-Dichloropropylene, and 1,1,2-Trichloroethane (see below for discussion of these three parameters). The results for the high spike (VOL C) had higher recoveries, ranging from 55-97% for many parameters. Some parameters appear to give problems, particularly Trichlorofluoromethane, 1,1-Dichloroethylene, and Carbon Tetrachloride.

The results for Tetrachloroethylene and 1,1,2,2-Tetrachloroethane were reported as less than ("<") a value. For the statistical calculations in Tables 1 and 2 (Appendix 1), and to calculate the percent recovery, half of the given value was used. The recovery for these two parameters is low relative to the design values, but this laboratory's results are semi-quantitative.

This laboratory reported that cis-1,2-Dichloropropylene, 1,1,2-Trichloroethane, and Dibromochloromethane co-eluted, and provided a combined result. One third of this value was entered into the spreadsheet for these parameters. These parameters are over-recovered relative to the design values, but this laboratory's results are semi-quantitative.

A result of 11.1 µg/L for Dichloromethane was reported in the blank (VOL A). This is a common laboratory contaminant and may be a problem for this participant. The result for Dichloromethane reported in the blank was not subtracted from the results for the spiked samples. The result for Dichloromethane in the low spike gave a recovery of 119%, which is higher than the recovery for the other parameters in the scan, except for 1,1,2-Trichloroethane and cis-1,3-Dichloropropylene (see above). The recovery for Dichloromethane in the high spike does not differ from the majority of the other parameters in the same manner. This suggests that the background level of Dichloromethane in Laboratory 1010 affects low level samples but does not have the same effect on samples with higher levels of volatile organics.

Excluding the specific parameters mentioned above, reasonably consistent performance across the scan was demonstrated. There was no specific pattern of recovery based on the order of gas chromatographic elution. There are several specific problem areas that need to be addressed by this laboratory, as noted above.

Laboratory 1012

This laboratory did not report results for Dichlorobromomethane and trans-1,3-Dichloropropylene. They used a 1% SP-1000 60/80 Carbopack packed column, with a jet separator. Schedule 3 of the MISA General Regulation (1) specifies the use of a capillary column. For regulatory purposes, this laboratory's results would not be acceptable. The results are reviewed for the purposes of improving laboratory performance.

The results for the spiked samples (VOL B and VOL C) demonstrated variable recovery relative to the design values. This was a more noticeable problem with the low spike, as the recovery ranged from 0-110% (excluding Chloromethane and Dichloromethane; see below). For the high spike, the recovery ranged from 9-95% (excluding Chloromethane and Dichloromethane).

A value of 9.1 µg/L was reported for Chloromethane. This is an unusual laboratory contaminant, due to the instability of this compound. This value was not subtracted from the spiked sample results, resulting in an apparently high recovery of this parameter in these samples. A value of 3.0 µg/L was reported for Dichloromethane. This is a common laboratory contaminant and may have contributed to the higher recovery of this parameter from the spiked samples. This value was not subtracted from the spiked sample results.

The recovery across the scan does not demonstrate any pattern (Figure 1). Specific parameters appear to present problems for this laboratory, such as Chloromethane (see above), Trichlorofluoromethane, 1,1-Dichloroethylene, Carbon Tetrachloride, and Tetrachloroethylene (low recovery of the latter four parameters). A similar pattern of performance was demonstrated in both spiked samples.

Laboratory 1013

This laboratory did not report results for 1,2-Dichloropropane, cis-1,3-Dichloropropylene, and trans-1,3-Dichloropropylene. They noted with their results that they could not explain the high result for Ethylene Dibromide in the high spike (VOL C), which they felt was out-of-line with the result from the other parameters. They used a SPB-5 60 m wide-bore, 0.32 mm I.D. capillary gas chromatographic column.

The results demonstrated good recovery relative to the design values, for most parameters, except for Ethylene Dibromide in the high spike. Carbon Tetrachloride and Tetrachloroethylene were under-recovered in both samples.

Nothing was reported in the blank sample (VOL A), so there is no evidence of any laboratory contamination problems.

Excluding the specific parameters mentioned above, reasonably consistent performance across the scan was demonstrated. There was no pattern of recovery based on the order of gas chromatographic elution. A similar pattern was demonstrated in both spiked samples.

Laboratory 1014

Laboratory 1014 included a note with their results indicating that they had had to repeat the analysis for Chloromethane and Bromomethane. The resulting headspace in the sample vials may have contributed to low results. After receiving the initial table of results, they reviewed their results for Dichloromethane and indicated that there was a background level of 9 µg/L. They requested that this amount be subtracted from the initial results reported for VOL A, VOL B, and VOL C. The corrected values were used to calculate the percent recovery and were plotted in Figures 3 and 6. They used a J & W DB-624 30 m fused silica capillary column.

The results demonstrated variable recovery relative to the design value. This was more pronounced in the low spike (VOL B), with recoveries varying from 0-140%. Recoveries from the high spike (VOL C) varied from 40-143%.

Results for the blank sample (VOL A) were all reported as less than the MDL. For most parameters, the results suggest that there was no background contamination in this laboratory, other than the level of Dichloromethane, noted above.

The recovery across the scan appears to cluster the parameters into three groups. The majority of compounds are consistently recovered, with values between 80-110% of the design value. The second group of parameters were under-recovered: Chloroform, Carbon Tetrachloride, trans-1,3-Dichloropropylene, and Tetrachloroethylene. This group of parameters also shows a decreasing pattern of recovery within the over-all scan, based on the order of gas chromatographic elution. The third group of parameters were over-recovered: Benzene, 1,1,2-Trichloroethane, and Ethylene Dibromide. This laboratory has several specific problem areas to be addressed as noted above.

4.2 Oil and Grease Samples

OVERVIEW OF INTERLABORATORY PERFORMANCE - OIL AND GREASE

The results from all of the participants for the two spiked samples (O&G B and O&G C) were plotted using Youden's two-sample technique (2) in Figure 10. A line was drawn between the expected values and the origin to indicate the target area of precision.

The results demonstrate that, regardless of solvent used, individual laboratories demonstrate systematic bias when analyzing for Oil and Grease. While none of the participants demonstrated a high bias (relative to the design value), several laboratories demonstrate a low bias. The results are not affected by random error, as the majority of the points are very close to the line.

To further demonstrate that the between-laboratory variability is more significant than the within-laboratory variability, the *F* Test may be used. After converting to percent recovery, the between-laboratory variance may be estimated from the sums of the spiked samples (O&G B and O&G C). The within-laboratory variance may be estimated from the differences of the spiked samples. The calculations given in Table III demonstrate that the between-laboratory variability is more significant then the within-laboratory variability.

**TABLE III - ESTIMATE OF BETWEEN-
VS WITHIN-LABORATORY VARIABILITY**

s^2_{Diff}	0.0063
s^2_{Sum}	0.1597
$F = \frac{s^2_{Sum}}{s^2_{Diff}}$	25.49

For $df_{Diff} = 11$, $df_{Sum} = 11$, and a 95% Confidence Interval, $F = 2.81$

The calculated *F* value is greater than 2.891, therefore the between-laboratory variability is significantly different from the within-laboratory variability.

There are several possible sources for the systematic error demonstrated by different participants. The method is sensitive to the timing of various steps in the analytical procedure (3). Variations in timing may cause different oils and greases to volatilize, resulting under-recovery. The purity of the solvent may affect the efficiency of the method. The temperature of the water bath also affects the recovery of the extracted materials.

The participants were not required to describe in detail their analytical procedure used for this interlaboratory study. It is desirable that the individual participants who have demonstrated systematic bias in this interlaboratory study, will strive to improve their performance. Future studies that include the analysis of Oil and Grease will attempt to monitor improvements in performance by the participants.

LABORATORY 1001

This laboratory used Freon for the extraction process. They had excellent recovery for both spiked samples (O&G B and O&G C), relative to the design values. However they appear to be biased high relative to the other participants who used the same solvent, both compared to the interlaboratory mean and median.

This laboratory also reported the highest level in the blank sample (O&G A). This suggests that there may be some background contamination in their laboratory. If the results for the spiked samples were corrected for the blank, the results would be closer to the interlaboratory mean and median.

The result for the effluent sample (O&G D) was also high, relative to the interlaboratory mean and median. This supports the above suggestion that there may be some background laboratory contamination.

LABORATORY 1002

This laboratory used Dichloromethane for the extraction process. They had excellent recovery for the spiked samples (O&G B and O&G C), relative to the design value. However they appear to be biased slightly high relative to the other participants, both for the interlaboratory mean and median.

There was no evidence of possible background contamination as there was nothing reported in the blank sample (O&G A).

The result for the effluent sample (O&G D) was also high relative to the interlaboratory mean and median (excluding the result from Laboratory 1004; see Table 3, Appendix 1). As there was only one result for this sample from each participant, it is not possible to ascertain whether the high result is due to laboratory bias, or sample matrix effects.

LABORATORY 1003

This laboratory used Dichloromethane for the extraction process. They had good recovery of the low spike (O&G B) but under-recovered the high spike (O&G C). As a result, they are in good agreement with the interlaboratory mean and median for the low spike but appear to be biased low for the high spike. This laboratory may need to improve its within-laboratory precision. Participation in future interlaboratory studies will allow for assessing improvement their performance.

There was no evidence of possible background contamination as there was nothing reported in the blank sample (O&G A).

The result for the effluent sample (O&G D) was low relative to the interlaboratory mean and median. As there was only one result for this sample from each participant, it is difficult to ascertain whether the low result is due to laboratory bias or the sample matrix.

LABORATORY 1004

Laboratory 1004 used Dichloromethane for the extraction process. Their results demonstrate good recovery relative to the design value for Sample O&G B and O&G C. They also demonstrate good agreement with the interlaboratory mean and median.

There was no evidence of possible background contamination as there was nothing reported in the blank sample (O&G A).

A very high result for the effluent sample was reported by this laboratory. Compared to the results from all of the other participants, regardless of solvent used, this result appears to be an anomaly. While every effort was made to provide a homogeneous effluent to all of the participants, this laboratory may have received an aliquot that contained some additional extractable materials. Laboratory contamination may also have contributed to the anomalous result. The statistical calculations in Table 3, Appendix 1 were performed with Laboratory 1004's result deleted.

LABORATORY 1007

This laboratory used Freon for the extraction process. They had excellent recovery for the spiked samples (O&G B and O&G C), relative to the design value. However they appear to be biased slightly high relative to the other participants, both for the interlaboratory mean and median.

There was no evidence of possible background contamination as there was nothing reported in the blank sample (O&G A).

The result for the effluent sample (O&G D) showed good agreement with the interlaboratory mean and median.

LABORATORY 1008

This laboratory used Freon for the extraction process. They demonstrated consistent within-laboratory precision for the two spiked samples (O&G B and O&G C) but under-recovered relative to the design values. The results are also low relative to the interlaboratory mean. However this laboratory reported the median values from all of the participants who used Freon for the extraction. While this laboratory demonstrates comparable performance with many of the participating laboratories, some effort should be made to improve accuracy.

There was no evidence of possible background contamination as there was nothing reported in the blank sample (O&G A).

The result for the effluent sample (O&G D) was low relative to the interlaboratory mean and median. As there was only one result for this sample from each participant, it is difficult to ascertain whether the low result is due to laboratory bias or the sample matrix.

LABORATORY 1009

This laboratory used Freon for the extraction process. They had good recovery of the low spike (O&G B) but under-recovered the high spike (O&G C). As a result, they are in good agreement with the interlaboratory mean and median for the low spike but appear to be biased low for the high spike. This laboratory may need to improve its within-laboratory precision. Participation in future interlaboratory studies will allow for assessing improvement their performance.

There was no evidence of possible background contamination as there was nothing reported in the blank sample (O&G A).

The result for the effluent sample (O&G D) was low relative to the interlaboratory mean and median. As there was only one result for this sample from each participant, it is difficult to ascertain whether the low result is due to laboratory bias or the sample matrix.

LABORATORY 1010

This laboratory used Freon for the extraction process. They demonstrated good within-laboratory precision for the two spiked samples (O&G B and O&G C), but had very low recovery relative to the design values. Their results are also biased very low relative to the interlaboratory mean and median. These results suggest that this laboratory is consistent in their performance of the analysis for Oil and Grease, but need to improve their method to produce more accurate results.

This laboratory also reported a result for the blank sample (O&G A). If this value had been subtracted from the spiked sample results, their low bias would have been even more pronounced.

The result for the effluent sample (O&G D) was comparable to the interlaboratory mean and median. As there was only one result for this sample from each participant, it is difficult to ascertain whether the laboratory performed better when analyzing this sample as compared to their results for the spiked reagent water samples (O&G B and O&G C).

LABORATORY 1011

This laboratory used Freon for the extraction process. They under-recovered the low spike (O&G B) but had good recovery of the high spike (O&G C). As a result, they are in good agreement with the interlaboratory mean and median for the high spike but are biased low for the low spike. This laboratory may need to improve its within-laboratory precision. Participation in future interlaboratory studies will allow for assessing improvement their performance.

There was no evidence of possible background contamination as there was nothing reported in the blank sample (O&G A). However the value represented as their lower detection limit (result was "<2 mg/L"), is higher than the MISA regulation MDL (for Group 25, MDL = 1000 µg/L, Ref. 1). Some effort should be made to improve the lower detection limit.

The result for the effluent sample (O&G D) showed good agreement with the interlaboratory mean and median.

LABORATORY 1013

This laboratory used Dichloromethane for the extraction process. They had low recovery relative to the design values for the spiked reagent water samples (O&G B and O&G C). Their results also appear to be biased low relative to the interlaboratory mean and median. The within-laboratory precision could be also be improved. Participation in future interlaboratory studies will allow for assessing improvement their performance.

There was no evidence of possible background contamination as there was nothing reported in the blank sample (O&G A).

The result for the effluent sample (O&G D) was comparable to the interlaboratory mean and median. As there was only one result for this sample from each participant, it is difficult to ascertain whether the laboratory performed better when analyzing this sample as compared to their results for the spiked reagent water samples (O&G B and O&G C).

LABORATORY 1014

This laboratory used Freon for the extraction process. They demonstrated good within-laboratory precision for the spiked reagent water samples (O&G B and O&G C). Their results are low relative to the design values. However they have good agreement with the interlaboratory mean and median. While this laboratory demonstrates comparable performance with many of the participating laboratories, some effort should be made to improve accuracy.

There was no evidence of possible background contamination as there was nothing reported in the blank sample (O&G A).

The result for the effluent sample (O&G D) was high relative to the interlaboratory mean and median. As there was only one result for this sample from each participant, it is not possible to ascertain whether the high result is due to any laboratory bias, or sample matrix effects.

LABORATORY 1015

This laboratory used Dichloromethane for the extraction process. They had excellent recovery for the spiked samples (O&G B and O&G C), relative to the design value. They have good agreement with the interlaboratory mean and median.

There was no evidence of possible background contamination as there was nothing reported in the blank sample (O&G A).

The result for the effluent sample (O&G D) was lower than the interlaboratory mean, but had good agreement with the interlaboratory median.

5 REFERENCES

1. ONTARIO REGULATION 695/88 as amended to Ontario Regulation 533/89 under the Environmental Protection Act; Effluent Monitoring - General

2. Youden, W. J. and Steiner, E.H.; 1975; Statistical Manual of the Association of Official Analytical Chemists; Association of Official Analytical Chemists; ISBN 0-935584-15-3

3. Standard Methods For the Examination of Water and Wastewater, 14th. Edition; 1976; American Public Health Association, American Water Works Association and The Water Pollution Control Federation; ISBN 0-87553-078-8

6 APPENDIX 1 - FULL DATA SET

Table 1	Volatiles, Samples VOL A, VOL B, and VOL C, Results in µg/L
Table 2	Volatiles, Samples VOL B and VOL C, Expressed as Percent Recovery of Design Value
Table 3	Oil and Grease, Samples O&G A, O&G B, O&G C, and O&G D, Results in mg/L
Table 4	Oil and Grease, Samples O&G B and O&G C, Results Expressed as Percent Recovery of Design Value
Figure 1	Volatiles, Labs 1001 and 1012
Figure 2	Volatiles, Sample VOL B, Labs 1002, 1003, and 1005
Figure 3	Volatiles, Sample VOL B, Labs 1007, 1009, 1013, and 1014
Figure 4	Volatiles, Lab 1010
Figure 5	Volatiles, Sample VOL C, Labs 1002, 1003, and 1005
Figure 6	Volatiles, Sample VOL C, Labs 1007, 1009, 1013, and 1014
Figure 7	Oil and Grease, Sample O&G B
Figure 8	Oil and Grease, Sample O&G C
Figure 9	Oil and Grease, Sample O&G D
Figure 10	Oil and Grease, Youden Plot of Samples O&G B and O&G C

TABLE 1 - INTERLABORATORY STUDY 89-1: VOLATILE ORGANICS (MISA GROUPS 16 & 17)
RESULTS EXPRESSED IN ppb

DISTRIBUTED: JANUARY 31, 1989		DESIGN	LABORATORY NUMBER													
SAMPLE	PARAMETER		1001	1002	1003	1005	1007	1009	1010	1012	1013	1014	MEAN	MEDIAN	MIN	MAX	STD DEV
VOLA	1,1,2,2-TETRACHLOROETHANE	0															
VOLA	1,1,2-TRICHLOROETHANE	0															
VOLA	1,1-DICHLOROETHANE	0															
VOLA	1,1-DICHLOROETHYLENE	0															
VOLA	1,2-DICHLOROBENZENE	0					0.9										
VOLA	1,2-DICHLOROETHANE	0															
VOLA	1,2-DICHLOROPROPANE	0															
VOLA	1,3-DICHLROBENZENE	0					0.7										
VOLA	1,4-DICHLOROBENZENE	0					0.5										
VOLA	BROMOFORM	0															
VOLA	BROMOMETHANE	0															
VOLA	CARBON TETRACHLORIDE	0															
VOLA	CHLOROBENZENE	0															
VOLA	CHLOROFORM	0															
VOLA	CHLOROMETHANE	0								9.1							
VOLA	cis-1,3-DICHLOROPROPYLENE	0															
VOLA	DICHLOROBROMOMETHANE	0															
VOLA	ETHYLENE DIBROMIDE	0															
VOLA	METHYLENE CHLORIDE	0		1.7					11.1	3							
VOLA	TETRACHLOROETHYLENE	0															
VOLA	trans-1,2-DICHLOROETHYLENE	0															
VOLA	trans-1,3-DICHLOROPROPYLENE	0															
VOLA	TRICHLOROETHYLENE	0															
VOLA	TRICHLOROFLUOROMETHANE	0	N/A														
VOLA	VINYL CHLORIDE	0															
VOLA	BENZENE	0															
VOLA	ETHYLBENZENE	0															
VOLA	TOLUENE	0						1.1									
VOLA	o-XYLENE	0															
VOLA	m-XYLENE	0															
VOLA	p-XYLENE	0															
VOLA	m- AND p-XYLENE	0		0.04													
VOLA	o- AND p-XYLENE	0															

* REPORTED AS CO-ELUTING WITH DIBROMOCHLOROMETHANE . SEE FOOTNOTE AT THE BOTTOM OF PAGE 1 REGARDING MDL ENTERED FOR EACH PARAMETER IN THE TABLE.

FOR RESULTS REPORTED AS " ", SEE AT THE REPORTED VALUE SEE PAGE FOR CONCENTRATION CALCULATIONS.

TABLE 1 - INTERLABORATORY STUDY 89-1: VOLATILE ORGANICS (MISA GROUPS 16 & 17)
RESULTS EXPRESSED IN ppb

DISTRIBUTED: JANUARY 31, 1989		DESIGN	LABORATORY NUMBER														
SAMPLE	PARAMETER		1001	1002	1003	1005	1007	1009	1010	1012	1013	1014	MEAN	MEDIAN	MIN	MAX	STD DEV
VOLB	1,1,2,2-TETRACHLOROETHANE	5	2	3.1	3.6	2.92	0.8	4.3	< 1.88	3.4	4	2.6	2.8	3.01	0.8	4.3	1.2
VOLB	1,1,2-TRICHLOROETHANE	5	4	5.7	7	5	1.3	4.7	* 10.9	3.8	3	6.8	5.2	4.8	1.3	10.9	2.6
VOLB	1,1-DICHLOROETHANE	5	8.7	4.8	3.7	3.56	0.2	4.4	3.32	0	4	5.7	3.8	3.85	0	8.7	2.5
VOLB	1,1-DICHLOROETHYLENE	5	15	3.2	3	2.39	0	3.4	1.09	0	7	6.6	4.2	3.1	0	15	4.5
VOLB	1,2-DICHLOROBENZENE	5	4.5	7.3	4.1	5.06	3.4	4.6	2.13	3.4	5	5.9	4.5	4.55	2.13	7.3	1.4
VOLB	1,2-DICHLOROETHANE	5	9.9	6.2	5.1	4.93	1.1	5.6		4.6	5	5.3	5.3	5.1	1.1	9.9	2.2
VOLB	1,2-DICHLOROPROPANE	5	7.1	5.2	4.4	4.24	2.7	4.4	1.92	3.9		6	4.4	4.4	1.92	7.1	1.6
VOLB	1,3-DICHLROBENZENE	5	4.4	6.8	4	4.87	3.4	4.4	1.82	3.6	7	5.4	4.6	4.4	1.82	7	1.6
VOLB	1,4-DICHLOROBENZENE	5	4.8	6.4	4.3	4.95	4.1	4.4	2.04	4.1	5	5.6	4.6	4.6	2.04	6.4	1.1
VOLB	BROMOFORM	5	3.6	5.1	5	5.03	2.1	4.4	1.35	3.6	5	5.5	4.1	4.7	1.35	5.5	1.4
VOLB	BROMOMETHANE	5	25	5.8	5.5	5.06	0	26		5.5	7	0	8.9	5.5	0	26	9.8
VOLB	CARBON TETRACHLORIDE	5	5.1	2.4	2.6	2.61	0	2.6	1.01	0.8	3	3.6	2.4	2.6	0	5.1	1.5
VOLB	CHLOROBENZENE	5	1.7	5	4.3	4.14	1.9	4	1.69	3.2	4	5	3.5	4	1.69	5	1.3
VOLB	CHLOROFORM	5	7.1	5	3.9	3.36	0.5	3.4	1.99	3.1	4	3.7	3.6	3.55	0.5	7.1	1.7
VOLB	CHLOROMETHANE	5	41	8.7	5.6	3.43	0	5.9		13	5	4.1	9.6	5.6	0	41	12.3
VOLB	cis-1,3-DICHLOROPROPYLENE	5	6	3.2	5.2	5.91	2.9	4.8	* 10.9	5.3		5.3	5.5	5.3	2.9	10.9	2.3
VOLB	DICHLOROBROMOMETHANE	5	7.3	5.2	5.1	4.43	2.6	4.9	2.11		5	5.8	4.7	5	2.11	7.3	1.6
VOLB	ETHYLENE DIBROMIDE	5	3.6	6.2	6.8	5.12	2.3	5.5	2.14	4.5	6	8.8	5.1	5.06	2.14	8.8	2.1
VOLB	METHYLENE CHLORIDE	5	2.6	6.3	3.8	4.54	0	2.1	5.95	8.8	4	5	4.3	4.27	0	8.8	2.5
VOLB	TETRACHLOROETHYLENE	5	2	3.2	5.7	1.83	2	2.5	< 1.73	1.5	3	3.3	2.6	2.25	1.5	5.7	1.3
VOLB	trans-1,2-DICHLOROETHYLENE	5	13	6	5	4.34	0	3.8	2.68	4.3	4	8.8	5.2	4.32	0	13	3.5
VOLB	trans-1,3-DICHLOROPROPYLENE	5	2	4.4	3.8	3.09	0	3				3.4	2.8	3.09	0	4.4	1.4
VOLB	TRICHLOROETHYLENE	5	7.3	6.1	4.4	5.13	0.4	3.8	2.07	3.2	4	7.2	4.4	4.2	0.4	7.3	2.2
VOLB	TRICHLOROFLUOROMETHANE	5		1.7	3	1.78	0	2.5	0.99	0	5	5.7	2.3	1.78	0	5.7	2.0
VOLB	VINYL CHLORIDE	5	32	5.9	5.6	3.16	0	4.1		5.1	4	7.2	7.5	5.1	0	32	9.4
VOLB	BENZENE	5	7.7	4.7	4.7	4.74	1.5	4.2	2.93	4.3	5	8.6	4.8	4.7	1.5	8.6	2.1
VOLB	ETHYLBENZENE	5	3.2	5.6	4.4	5	2.8	4.7	2.01	4	5	5.8	4.3	4.55	2.01	5.8	1.2
VOLB	TOLUENE	5	2.9	5.2	4.9	4.63	3.7	3.3	2.03	3.6	3	7.3	4.1	3.65	2.03	7.3	1.5
VOLB	o-XYLENE	5	3	5.8	4.8	5.04	5.5	5.2		3.9	5	6	4.9	5.02	3	6	0.9
VOLB	m-XYLENE	5	3						2.17	3.4			2.9	3	2.17	3.4	0.6
VOLB	p-XYLENE	5	3							3.9							
VOLB	m- AND p-XYLENE	10		5.8	0.9	8.65	4.6	9.5			9	10.4	8.1	8.9	4.6	10.4	2.1
VOLB	o- AND p-XYLENE	10							4.02								

* REPORTED AS CO-ELUTING WITH DIBROMOCHLOROMETHANE. ONE HALF OF THE ABOVE VALUE WAS USED IN CALCULATING THE MEAN FOR EACH PARAMETER IN THE TABLE.

FOR RESULTS REPORTED AS "<", ONE HALF OF THE REPORTED VALUE WAS USED FOR CALCULATION PURPOSES.

TABLE 1 - INTERLABORATORY STUDY 89-1: VOLATILE ORGANICS (MISA GROUPS 16 & 17)
RESULTS EXPRESSED IN ppb

DISTRIBUTED: JANUARY 31, 1989		DESIGN	LABORATORY NUMBER														
SAMPLE	PARAMETER		1001	1002	1003	1005	1007	1009	1010	1012	1013	1014	MEAN	MEDIAN	MIN	MAX	STD DEV
VOLC	1,1,2,2-TETRACHLOROETHANE	20	!BT	13.6	16.7	14.42	7.9	18.1	< 11.9	13	13	17.1	13.3	13.6	7.9	18.1	4.1
VOLC	1,1,2-TRICHLOROETHANE	20	!BT	22.4	33.5	15.84	12.2	14.4	*107.6	17	12	24.7	28.8	17	12	107.6	30.3
VOLC	1,1-DICHLOROETHANE	20	!BT	17.9	14.2	11.11	6.9	10.9	17.8	9	12	17.7	13.1	12	6.9	17.9	4.1
VOLC	1,1-DICHLOROETHYLENE	20	!BT	12	10.6	7.61	2.2	7.2	5.55	3.1	12	16.2	8.5	7.61	2.2	16.2	4.6
VOLC	1,2-DICHLOROBENZENE	20	!BT	26.2	16.5	21.24	14.5	15.3	15.9	14	18	21.5	18.1	16.5	14	26.2	4.1
VOLC	1,2-DICHLOROETHANE	20	!BT	23.6	21.3	21.*	10.2	16.5		17	16	27.8	19.2	19.15	10.2	27.8	5.4
VOLC	1,2-DICHLOROPROPANE	20	!BT	20.4	17.6	13.84	11.9	11.5	12.3	14		19.9	15.2	13.92	11.5	20.4	3.6
VOLC	1,3-DICHLROBENZENE	20	!BT	23.9	15	17.51	14.2	13.3	13.4	13	25	20.2	17.3	15	13	25	4.7
VOLC	1,4-DICHLOROBENZENE	20	!BT	22.8	16.7	23.82	13.7	13.4	14.8	11	16	22	17.1	16	11	23.82	4.6
VOLC	BROMOFORM	20	!BT	22.4	22.2	16.74	12.5	18.3	13	17	17	22.9	18.0	17	12.5	22.9	3.9
VOLC	BROMOMETHANE	20	!BT	23.5	20.3	10.38	8.4	63.3		16	19	8.3	21.1	17.5	8.3	63.3	18.0
VOLC	CARBON TETRACHLORIDE	20	!BT	8.7	9.1	7.73	2.6	5.8	6.26	3	7	10.9	6.8	7	2.6	10.9	2.7
VOLC	CHLOROBENZENE	20	!BT	18	17.7	12.18	10.9	9	11.3	10	14	17.6	13.4	12.18	9	18	3.5
VOLC	CHLOROFORM	20	!BT	18.1	15.5	11.96	6.7	8.4	13.9	10	15	13.6	12.6	13.6	6.7	18.1	3.7
VOLC	CHLOROMETHANE	20	!BT	30.5	20.7	5.72	15.1	12.9		22	17	14.1	17.3	16.05	5.72	30.5	7.3
VOLC	cis-1,3-DICHLOROPROPYLENE	20	!BT	17.2	23.8	15.08	14.1	14.6	*107.4	17		26.6	29.5	17.1	14.1	107.4	31.8
VOLC	DICHLOROBROMOMETHANE	20	!BT	21.4	21.2	13.89	12.2	13.8	16.9		17	25.2	17.7	16.95	12.2	25.2	4.5
VOLC	ETHYLENE DIBROMIDE	20	!BT	25.6	29.8	16.8	11.8	18.5	18.8	19	120	28.5	32.1	19	11.8	120	33.5
VOLC	METHYLENE CHLORIDE	20	!BT	21.2	14.7	12.83	1.2	4.5	19.2	20	20	21	15.0	19.2	1.2	21.2	7.5
VOLC	TETRACHLOROETHYLENE	20	!BT	10.7	22.8	5.44	5.7	5.8	< 11	5	8	7.9	8.5	7.9	5	22.8	5.7
VOLC	trans-1,2-DICHLOROETHYLENE	20	!BT	22.2	18	10.34	2.5	10.3	11.7	9	14	22.3	13.4	11.7	2.5	22.3	6.5
VOLC	trans-1,3-DICHLOROPROPYLENE	20	!BT	17.5	18.2	11.28	5.2	8.5				13.8	12.4	12.54	5.2	18.2	5.1
VOLC	TRICHLOROETHYLENE	20	!BT	22	16.8	11.12	8.2	8.3	12.7	11	18	17.9	14.0	12.7	8.2	22	4.8
VOLC	TRICHLOROFLUOROMETHANE	20	!BT	6.9	9.8	5.12	3.1	5.2	4.87	1.7	10	11.8	6.5	5.2	1.7	11.8	3.4
VOLC	VINYL CHLORIDE	20	!BT	22.4	19.7	7.12	10.1	7.1		6	13	18.8	13.0	11.55	6	22.4	6.5
VOLC	BENZENE	20	!BT	20.1	18.3	16.74	11.7	9.7	19.3	12	17	26.6	16.8	17	9.7	26.6	5.2
VOLC	ETHYLBENZENE	20	!BT	20.9	17.2	15.28	3.5	11.1	12.6	11	14	19	13.8	14	3.5	20.9	5.2
VOLC	TOLUENE	20	!BT	21	21.4	10.61	11.1	6.9	12.9	13	18	19.8	15.0	13	6.9	21.4	5.2
VOLC	o-XYLENE	20	!BT	22.7	19.4	23.29	12.5	13.8		12	17	20	17.6	18.2	12	23.29	4.5
VOLC	m-XYLENE	20	!BT						13.2	12							
VOLC	p-XYLENE	20	!BT							12							
VOLC	m- AND p-XYLENE	40		21.7	33.2	24.1	4.6	20.1			28	34	29.7	42.1	4.6	34	10.0
VOLC	o- AND p-XYLENE	40							26								

!BT - SAMPLE BROKEN IN TRANSIT

* REPORTED AS CO-ELUTING WITH DIBROMOCHLOROMETHANE. ONE HALF OF THE DATA VALUE REPORTED WAS ENTERED FOR EACH PARAMETER IN THE TABLE.

FOR RESULTS REPORTED AS "<", HALF OF THE REPORTED VALUE WAS USED FOR STATISTICAL CALCULATIONS.

TABLE 3 - INTERLABORATORY STUDY 89-1: OIL AND GREASE
RESULTS EXPRESSSED AS mg/L (MISA GROUP 25)

DISTRIBUTED: JANUARY 31, 1989		LABORATORY NUMBER											
SAMPLE	DESIGN	1001	1002	1003	1004	1007	1008	1009	1010	1011	1013	1014	1015
O&GA	0	2	<1	<1	<1	<1	0	<1	1	<2	0	0.2	<1
O&GB	5	5	5.5	4	4	5.2	3.6	4.1	2.2	3	2.4	3.57	5
O&GC	20	21	20.3	11	18.5	21	14.5	13.8	9.1	16	12.3	13.97	18
O&GD		12	18	7	112.5	7	3.8	4.5	6.6	7	12.3	12.28	8
SOLVENT USED FOR EXTRACTION:		F	D	D	D	F	F	F	F	F	D	F	D

LEGEND: F - FREON; D - DICHLOROMETHANE

SOLVENT: FREON												
SAMPLE	DESIGN	1001	1007	1008	1009	1010	1011	1014	MEAN	STD DEV	MIN	MAX	MEDIAN
O&GA	0	2	0.5	0	0.5	1	1	0.2	0.7	0.7	0.0	2.0	0.5
O&GB	5	5	5.2	3.6	4.1	2.2	3	3.57	3.8	1.1	2.2	5.2	3.6
O&GC	20	21	21	14.5	13.8	9.1	16	13.97	15.6	4.2	9.1	21.0	14.5
O&GD		12	7	3.8	4.5	6.6	7	12.28	7.6	3.3	3.8	12.3	7

SOLVENT: DICHLOROMETHANE											
SAMPLE	DESIGN	1002	1003	1004	1013	1015	MEAN	STD DEV	MIN	MAX	MEDIAN
O&GA	0	0.5	0.5	0.5	0	0.5	0.4	0.2	0	0.5	0.5
O&GB	5	5.5	4	4	2.4	5	4.2	1.2	2.4	5.5	4
O&GC	20	20.3	11	18.5	12.3	18	16.0	4.1	11	20.3	18
O&GD *		18	7	112.5	12.3	8	11.3	5.0	7	18	10.15

* LABORATORY 1004 RESULT EXCLUDED FROM STATISTICAL CALCULATIONS.

NOTE: RESULTS WERE DIVIDED INTO TWO GROUPS, DEPENDING ON SOLVENT USED FOR EXTRACTION. STATISTICAL CALCULATIONS DONE ON SEPARATE DATA SETS ONLY. SEE SECTION 3.4 REGARDING ADJUSTMENT OF RESULTS REPORTED "<".

TABLE 2 - INTERLABORATORY STUDY 89-1: VOLATILE ORGANICS (MISA GROUPS 16 & 17)
RESULTS EXPRESSED AS PERCENT RECOVERY OF DESIGN VALUE

DISTRIBUTED: JANUARY 31, 1989		DESIGN	LABORATORY NUMBER														
SAMPLE	PARAMETER	(ppb)	1001	1002	1003	1005	1007	1009	1010	1012	1013	1014	MEAN	MEDIAN	MIN	MAX	STD DEV
VOLB	1,1,2,2-TETRACHLOROETHANE	5	40%	62%	72%	58%	16%	86%	19%	68%	80%	52%	55%	60%	16%	86%	24%
VOLB	1,1,2-TRICHLOROETHANE	5	80%	114%	140%	100%	26%	94%	218%	76%	60%	136%	104%	97%	26%	218%	53%
VOLB	1,1-DICHLOROETHANE	5	174%	96%	74%	71%	4%	88%	66%	0%	80%	114%	77%	102%	0%	174%	50%
VOLB	1,1-DICHLOROETHYLENE	5	300%	64%	60%	48%	0%	68%	22%	0%	140%	132%	83%	62%	0%	300%	90%
VOLB	1,2-DICHLOROBENZENE	5	90%	146%	82%	101%	68%	92%	43%	68%	100%	118%	91%	91%	43%	146%	29%
VOLB	1,2-DICHLOROETHANE	5	198%	124%	102%	99%	22%	112%		92%	100%	106%	106%	77%	22%	198%	45%
VOLB	1,2-DICHLOROPROPANE	5	142%	104%	88%	85%	54%	88%	38%	78%		120%	80%	88%	0%	142%	41%
VOLB	1,3-DICHLROBENZENE	5	88%	136%	80%	97%	68%	88%	36%	72%	140%	108%	91%	88%	36%	140%	31%
VOLB	1,4-DICHLOROBENZENE	5	96%	128%	86%	99%	82%	88%	41%	82%	100%	112%	91%	92%	41%	128%	23%
VOLB	BROMOFORM	5	72%	102%	100%	101%	42%	88%	27%	72%	100%	110%	81%	94%	27%	110%	28%
VOLB	BROMOMETHANE	5	500%	116%	110%	101%	0%	520%		110%	140%	0%	177%	110%	0%	520%	195%
VOLB	CARBON TETRACHLORIDE	5	102%	48%	52%	52%	0%	52%	20%	16%	60%	72%	47%	52%	0%	102%	29%
VOLB	CHLOROBENZENE	5	34%	100%	86%	83%	38%	80%	34%	64%	80%	100%	70%	80%	34%	100%	26%
VOLB	CHLOROFORM	5	142%	100%	78%	67%	10%	68%	40%	62%	80%	74%	72%	71%	10%	142%	35%
VOLB	CHLOROMETHANE	5	820%	174%	112%	69%	0%	118%		260%	100%	82%	193%	112%	0%	820%	246%
VOLB	cis-1,3-DICHLOROPROPYLENE	5	120%	64%	104%	118%	58%	96%	218%	106%		106%	99%	106%	0%	218%	56%
VOLB	DICHLOROBROMOMETHANE	5	146%	104%	102%	89%	52%	98%	42%		100%	116%	94%	100%	42%	146%	31%
VOLB	ETHYLENE DIBROMIDE	5	72%	124%	136%	102%	46%	110%	43%	90%	120%	176%	102%	106%	43%	176%	41%
VOLB	DICHLOROMETHANE	5	52%	126%	76%	91%	0%	42%	119%	176%	80%	100%	104%	86%	0%	280%	79%
VOLB	TETRACHLOROETHYLENE	5	40%	64%	114%	37%	40%	50%	17%	30%	60%	66%	52%	45%	17%	114%	27%
VOLB	trans-1,2-DICHLOROETHYLENE	5	260%	120%	100%	87%	0%	76%	54%	86%	80%	176%	104%	87%	0%	260%	71%
VOLB	trans-1,3-DICHLOROPROPYLENE	5	40%	88%	76%	62%	0%	60%				68%	49%	62%	0%	88%	33%
VOLB	TRICHLOROETHYLENE	5	146%	122%	88%	103%	8%	76%	41%	64%	80%	144%	87%	84%	8%	146%	44%
VOLB	TRICHLOROFLUOROMETHANE	5		34%	60%	36%	0%	50%	20%	0%	100%	114%	46%	36%	0%	114%	40%
VOLB	VINYL CHLORIDE	5	640%	118%	112%	63%	0%	82%		102%	80%	144%	149%	102%	0%	640%	189%
VOLB	BENZENE	5	154%	94%	94%	95%	30%	84%	59%	86%	100%	172%	97%	94%	30%	172%	41%
VOLB	ETHYLBENZENE	5	64%	112%	88%	100%	56%	94%	40%	80%	100%	116%	85%	91%	40%	116%	25%
VOLB	TOLUENE	5	58%	104%	98%	93%	74%	66%	41%	72%	60%	146%	81%	73%	41%	146%	30%
VOLB	o-XYLENE	5	60%	116%	96%	101%	110%	104%		78%	100%	120%	98%	101%	60%	120%	19%
VOLB	m-XYLENE	5	60%						43%	68%			57%	60%	43%	68%	13%
VOLB	p-XYLENE	5	60%							78%							
VOLB	m- AND p-XYLENE	10		58%	89%	87%	46%	95%			90%	104%	81%	89%	46%	104%	21%
VOLB	o- AND p-XYLENE	10							40%								

TABLE 2 - INTERLABORATORY STUDY 89-1: VOLATILE ORGANICS (MISA GROUPS 16 & 17)
RESULTS EXPRESSED AS PERCENT RECOVERY OF DESIGN VALUE

DISTRIBUTED: JANUARY 31, 1989 SAMPLE PARAMETER		DESIGN (ppb)	LABORATORY NUMBER 1001	1002	1003	1005	1007	1009	1010	1012	1013	1014	MEAN	MEDIAN	MIN	MAX	STD DEV
VOLC	1,1,2,2-TETRACHLOROETHANE	20		68%	84%	72%	40%	91%	30%	65%	65%	86%	67%	68%	30%	91%	20%
VOLC	1,1,2-TRICHLOROETHANE	20		112%	168%	79%	61%	72%	538%	85%	60%	124%	144%	85%	60%	538%	152%
VOLC	1,1-DICHLOROETHANE	20		90%	71%	56%	35%	55%	89%	45%	60%	89%	65%	60%	35%	90%	20%
VOLC	1,1-DICHLOROETHYLENE	20		60%	53%	38%	11%	36%	28%	16%	60%	81%	42%	38%	11%	81%	23%
VOLC	1,2-DICHLOROBENZENE	20		131%	83%	106%	73%	77%	80%	70%	90%	108%	91%	84%	70%	131%	20%
VOLC	1,2-DICHLOROETHANE	20		118%	107%	107%	51%	83%		85%	80%	139%	96%	96%	51%	139%	27%
VOLC	1,2-DICHLOROPROPANE	20		102%	88%	69%	60%	58%	62%	70%		100%	67%	70%	0%	102%	30%
VOLC	1,3-DICHLROBENZENE	20		119%	75%	88%	71%	67%	67%	65%	125%	101%	86%	75%	65%	125%	23%
VOLC	1,4-DICHLOROBENZENE	20		114%	84%	119%	69%	67%	74%	55%	80%	110%	86%	80%	55%	119%	23%
VOLC	BROMOFORM	20		112%	111%	84%	63%	92%	65%	85%	85%	115%	90%	85%	63%	115%	19%
VOLC	BROMOMETHANE	20		118%	102%	52%	42%	317%		80%	95%	42%	106%	88%	42%	317%	90%
VOLC	CARBON TETRACHLORIDE	20		44%	46%	39%	13%	29%	31%	15%	35%	55%	34%	35%	13%	55%	14%
VOLC	CHLOROBENZENE	20		90%	89%	61%	55%	45%	57%	50%	70%	88%	67%	61%	45%	90%	18%
VOLC	CHLOROFORM	20		91%	78%	60%	34%	42%	70%	50%	75%	68%	63%	60%	34%	91%	18%
VOLC	CHLOROMETHANE	20		153%	104%	29%	76%	65%		110%	85%	71%	86%	81%	29%	153%	37%
VOLC	cis-1,3-DICHLOROPROPYLENE	20		86%	119%	75%	71%	73%	537%	85%		133%	131%	86%	0%	537%	157%
VOLC	DICHLOROBROMOMETHANE	20		107%	106%	69%	61%	69%	85%		85%	126%	88%	85%	61%	126%	23%
VOLC	ETHYLENE DIBROMIDE	20		128%	149%	84%	59%	93%	94%	95%	600%	143%	160%	95%	59%	600%	167%
VOLC	DICHLOROMETHANE	20		106%	74%	64%	6%	23%	96%	100%	100%	105%	80%	96%	6%	150%	44%
VOLC	TETRACHLOROETHYLENE	20		53%	114%	27%	29%	29%	28%	25%	40%	40%	43%	29%	25%	114%	28%
VOLC	trans-1,2-DICHLOROETHYLENE	20		111%	90%	52%	13%	52%	59%	45%	70%	112%	67%	59%	13%	112%	33%
VOLC	trans-1,3-DICHLOROPROPYLENE	20		88%	91%	56%	26%	43%				69%	53%	63%	0%	91%	33%
VOLC	TRICHLOROETHYLENE	20		110%	84%	56%	41%	42%	64%	55%	90%	90%	70%	64%	41%	110%	24%
VOLC	TRICHLOROFLUOROMETHANE	20		35%	49%	26%	16%	26%	24%	9%	50%	59%	32%	26%	9%	59%	17%
VOLC	VINYL CHLORIDE	20		112%	99%	36%	51%	36%		30%	65%	94%	65%	58%	30%	112%	32%
VOLC	BENZENE	20		101%	92%	84%	59%	49%	97%	60%	85%	133%	84%	85%	49%	133%	26%
VOLC	ETHYLBENZENE	20		105%	86%	76%	18%	56%	63%	55%	70%	95%	69%	70%	18%	105%	26%
VOLC	TOLUENE	20		105%	107%	53%	56%	35%	65%	65%	90%	99%	75%	65%	35%	107%	26%
VOLC	o-XYLENE	20		114%	97%	116%	63%	69%		60%	85%	100%	88%	91%	60%	116%	22%
VOLC	m-XYLENE	20							66%	60%							
VOLC	p-XYLENE	20								60%							
VOLC	m- AND p-XYLENE	40		54%	83%	60%	12%	50%			70%	85%	59%	60%	12%	85%	25%
VOLC	o- AND p-XYLENE	40							65%								

TABLE 4 - INTERLABORATORY STUDY 89-1: OIL AND GREASE (MISA GROUP 25)
RESULTS EXPRESSSED AS PERCENT RECOVERY OF DESIGN VALUE

DISTRIBUTED: JANUARY 31, 1989

SOLVENT: FREON

SAMPLE	DESIGN	LABORATORY NUMBER 1001	1007	1008	1009	1010	1011	1014	MEAN	STD DEV	MIN	MAX	MEDIAN
O&GB	5	100%	104%	72%	82%	44%	60%	71%	76%	21%	44%	104%	72%
O&GC	20	105%	105%	73%	69%	46%	80%	70%	78%	21%	46%	105%	73%

SOLVENT: DICHLOROMETHANE

SAMPLE	DESIGN	1002	1003	1004	1013	1015	MEAN	STD DEV	MIN	MAX	MEDIAN
O&GB	5	110%	80%	80%	48%	100%	84%	24%	48%	110%	80%
O&GC	20	102%	55%	93%	62%	90%	80%	21%	55%	102%	90%

NOTE: RESULTS WERE DIVIDED INTO TWO GROUPS, DEPENDING ON SOLVENT USED FOR EXTRACTION. ABOVE STATISTICAL CALCULATIONS DONE ON SEPARATE DATA SETS ONLY.

FIGURE 1 – INTERLABORATORY STUDY 89–1

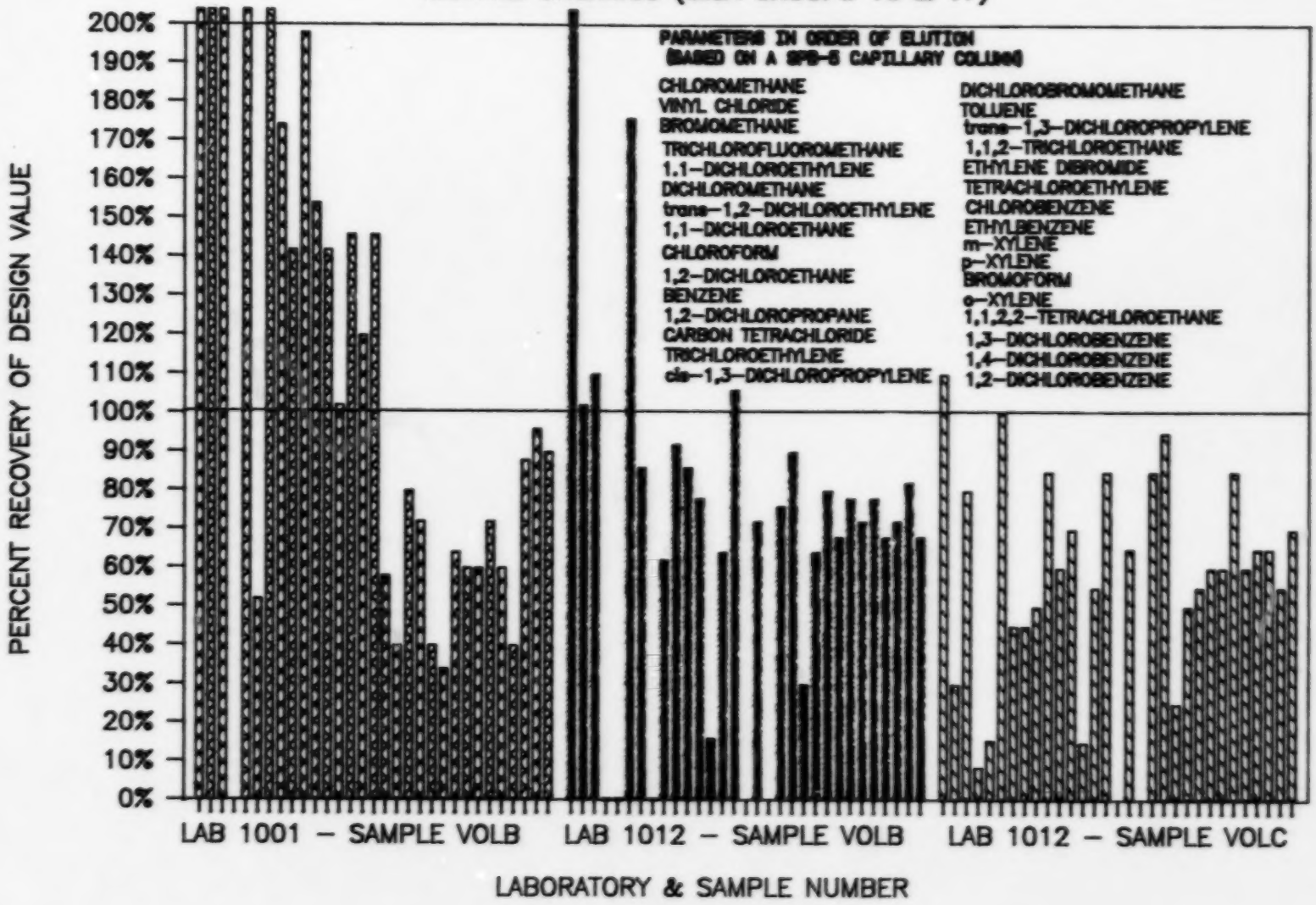


FIGURE 2 – INTERLABORATORY STUDY 89–1

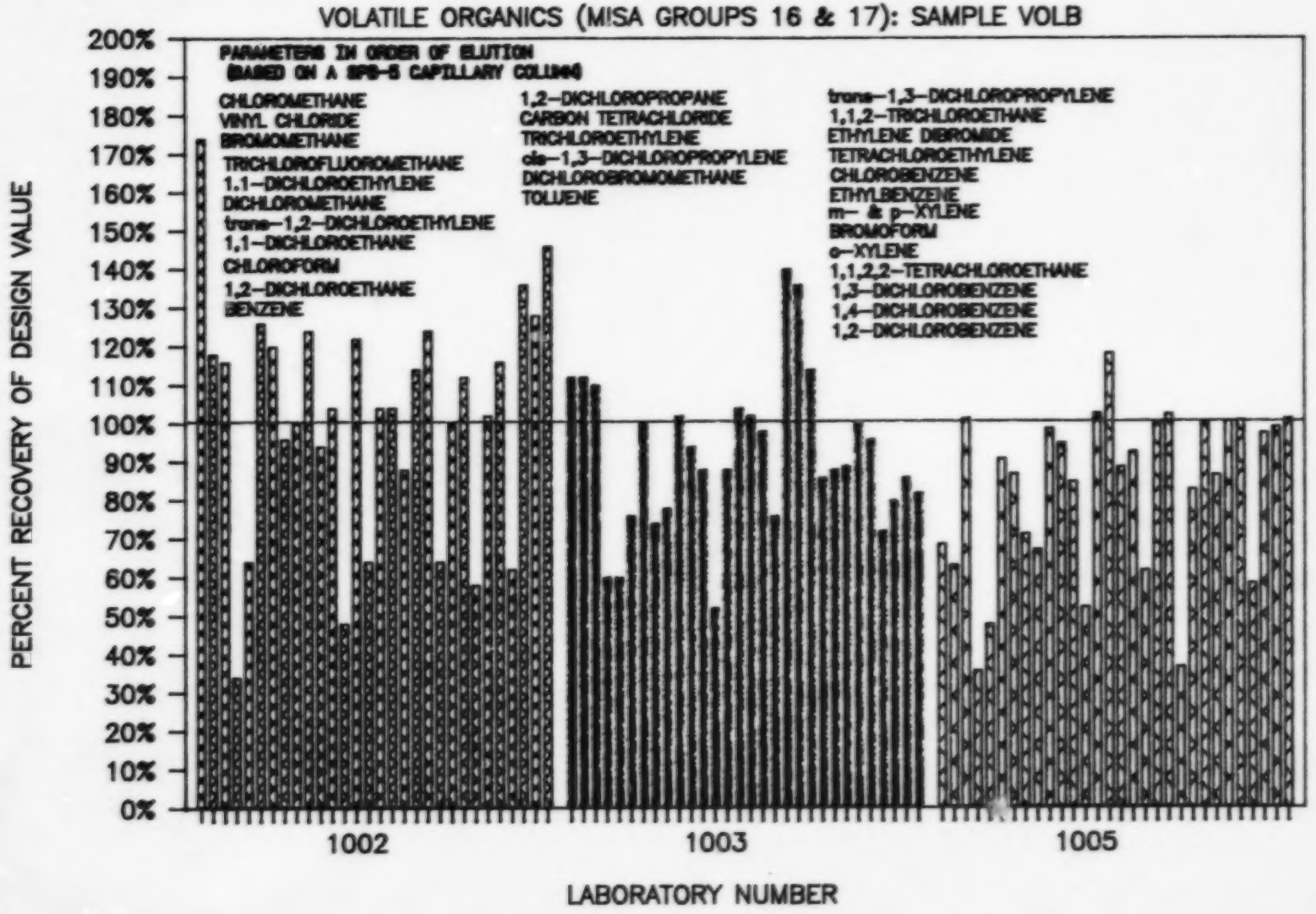


FIGURE 3 – INTERLABORATORY STUDY 89–1

VOLATILE ORGANICS (MISA GROUPS 16 & 17): SAMPLE VOLB

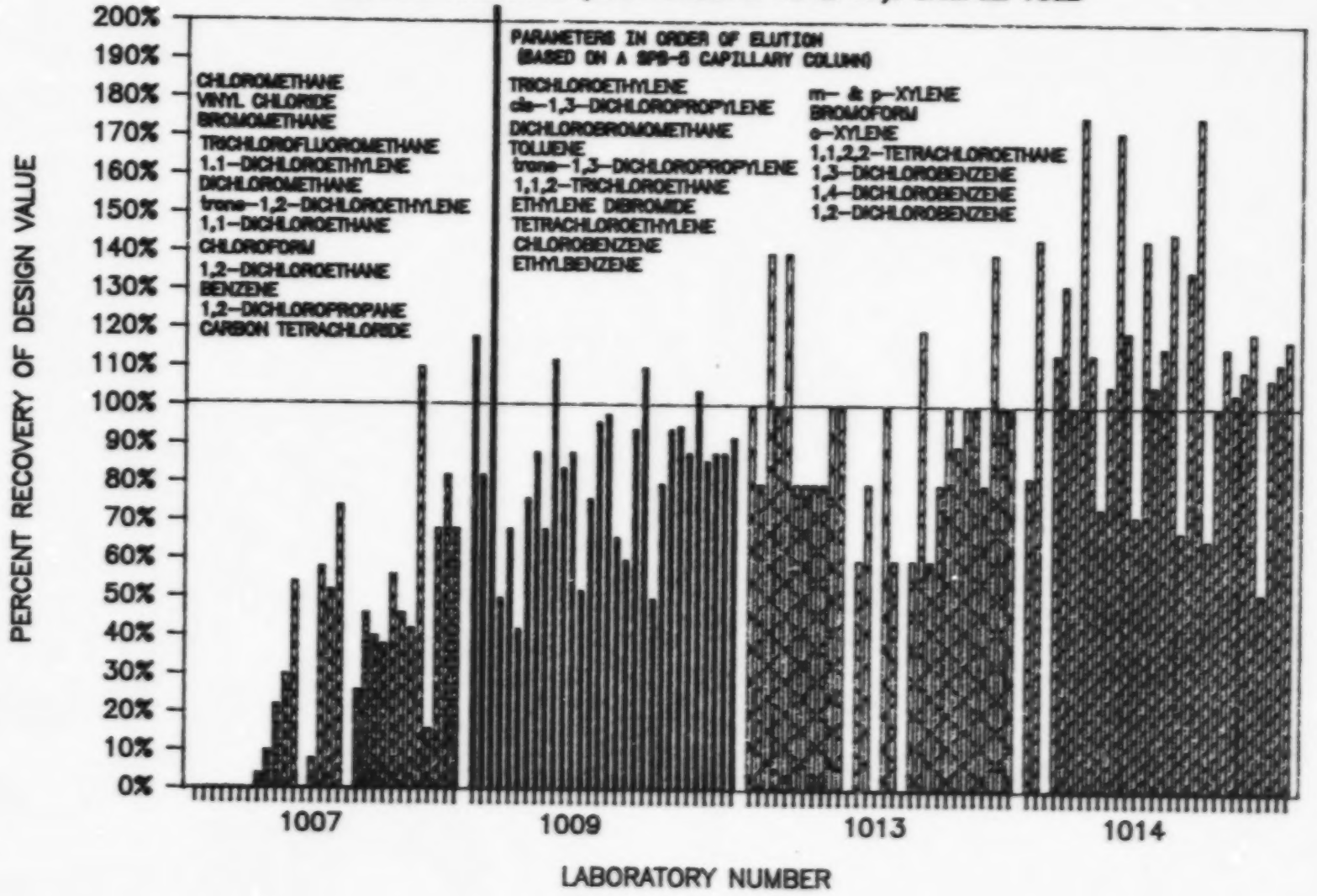


FIGURE 4 – INTERLABORATORY STUDY 89–1

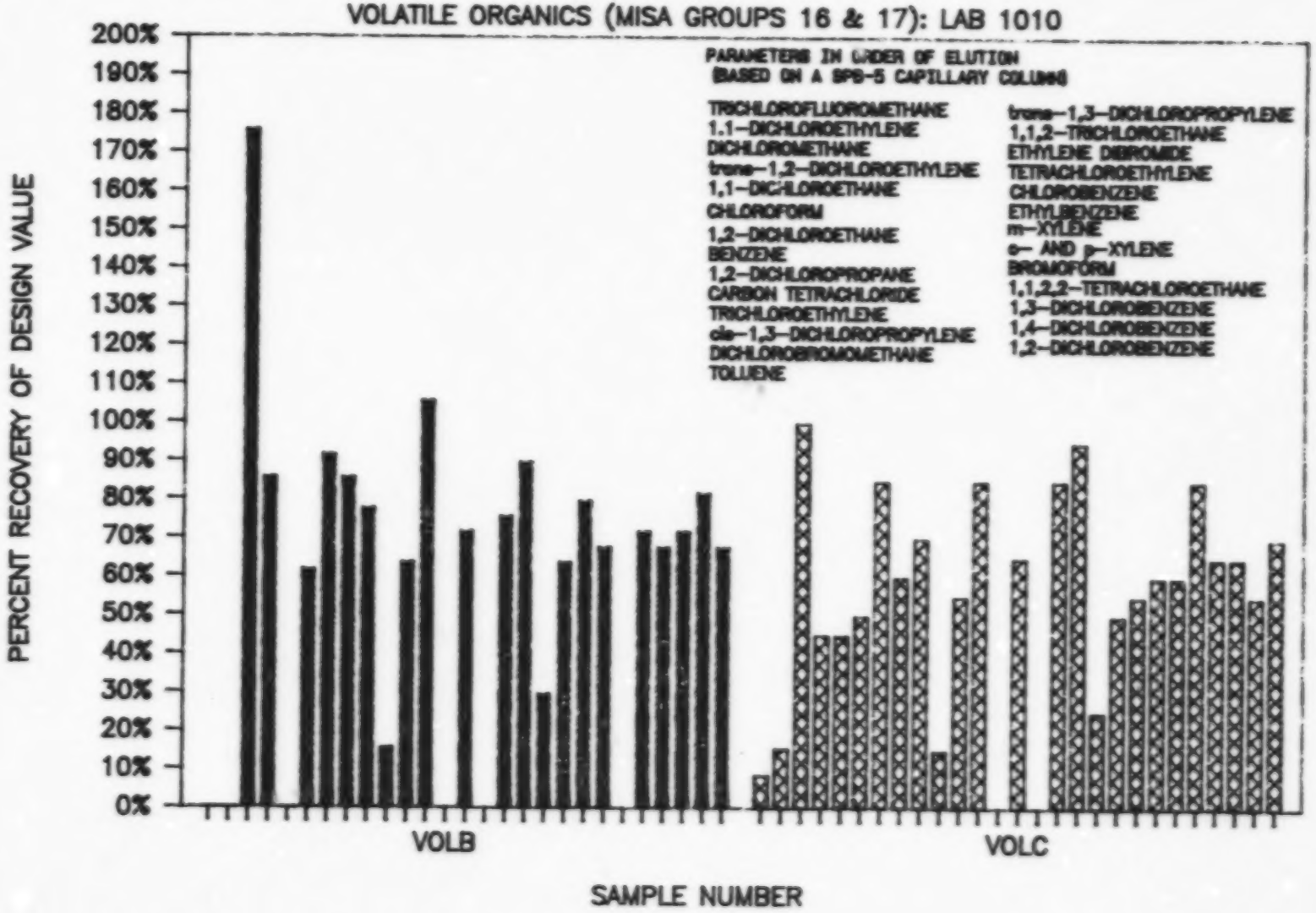


FIGURE 5 – INTERLABORATORY STUDY 89–1

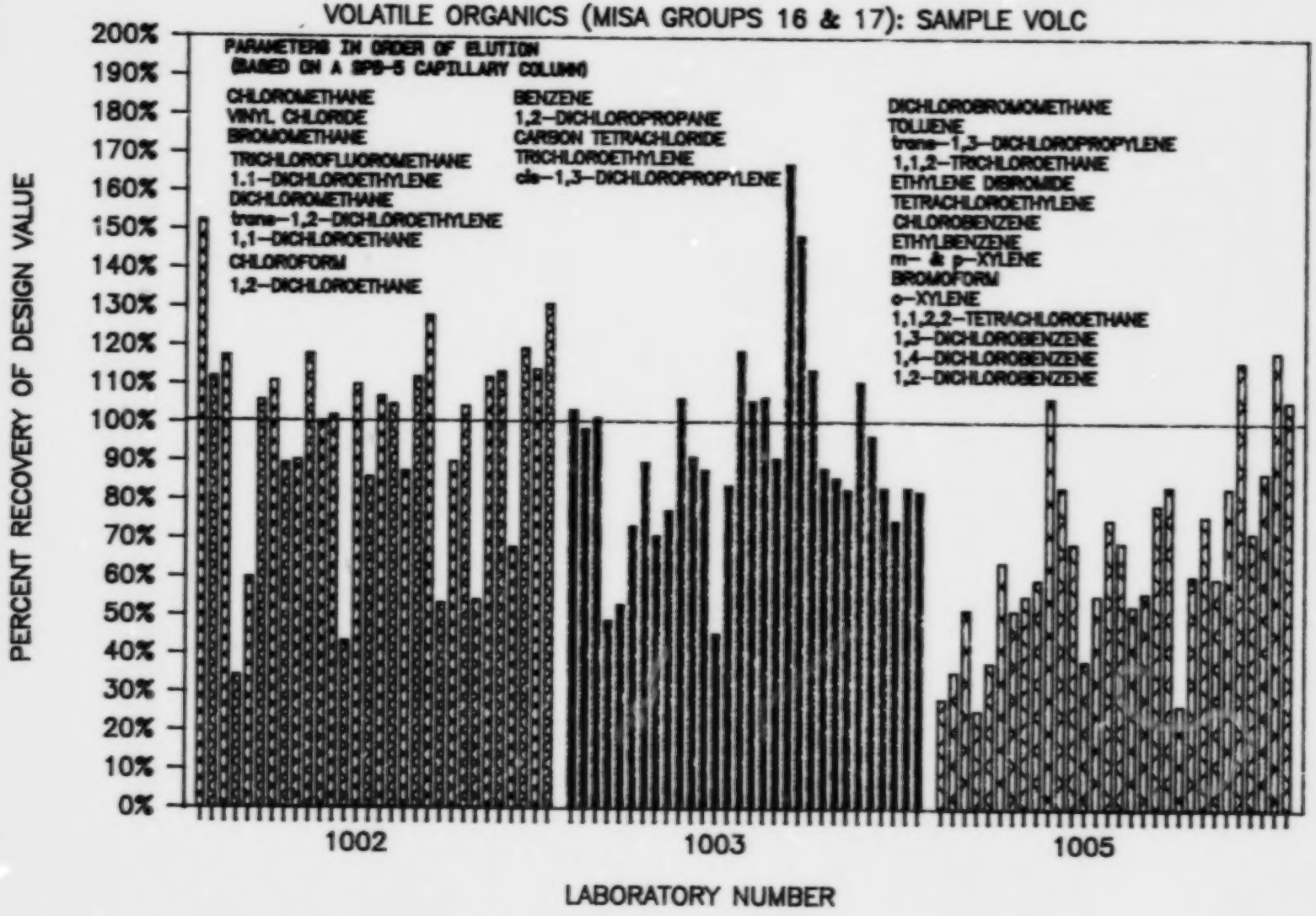


FIGURE 6 – INTERLABORATORY STUDY 89–1

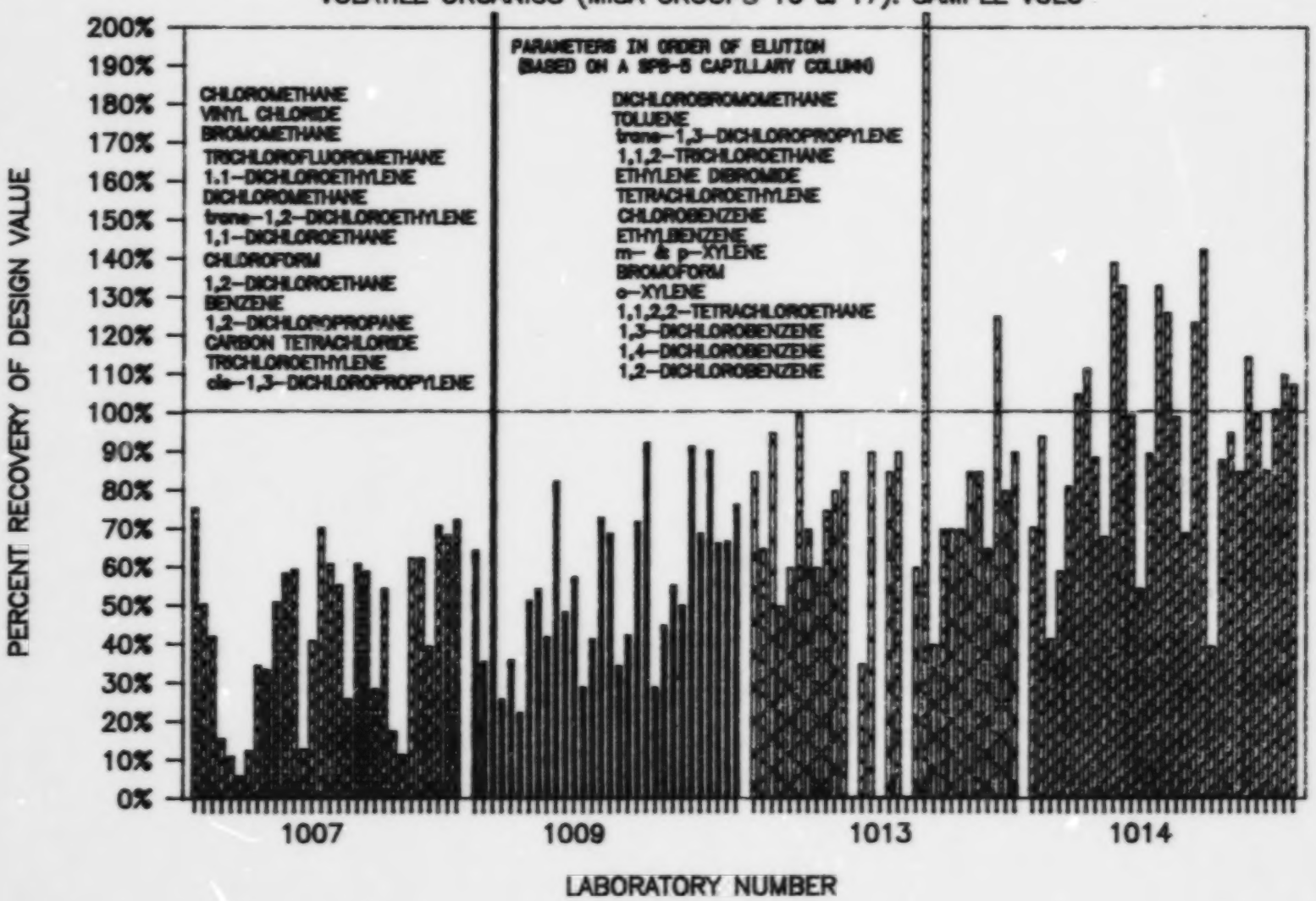


FIGURE 7 – INTERLABORATORY STUDY 89-1

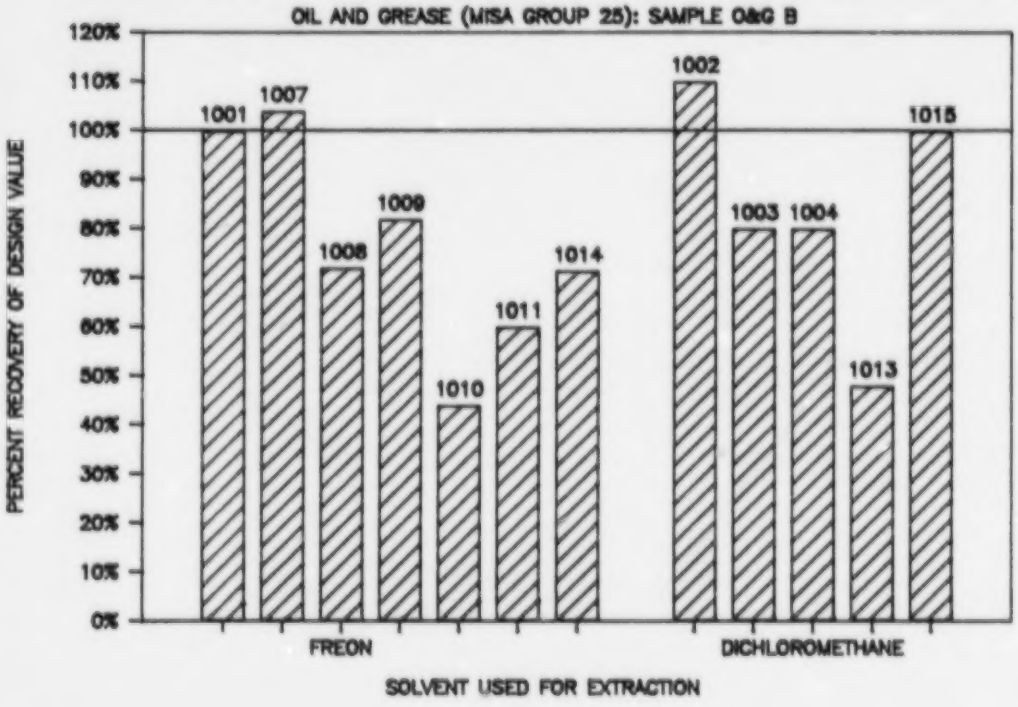


FIGURE 8 – INTERLABORATORY STUDY 89-1

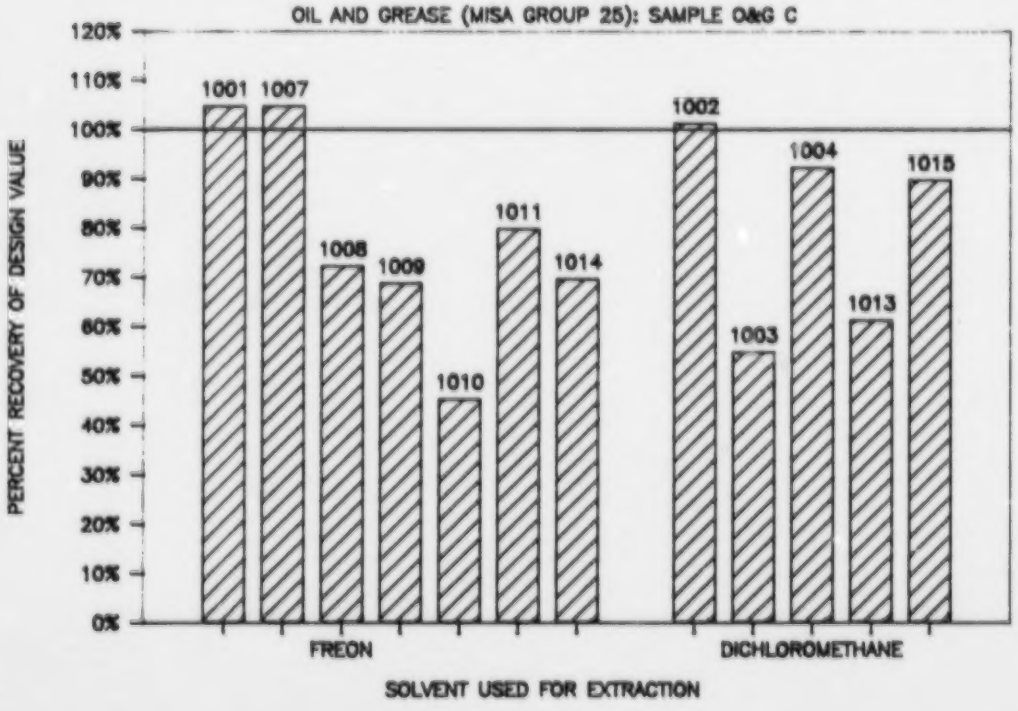


FIGURE 9 – INTERLABORATORY STUDY 89–1

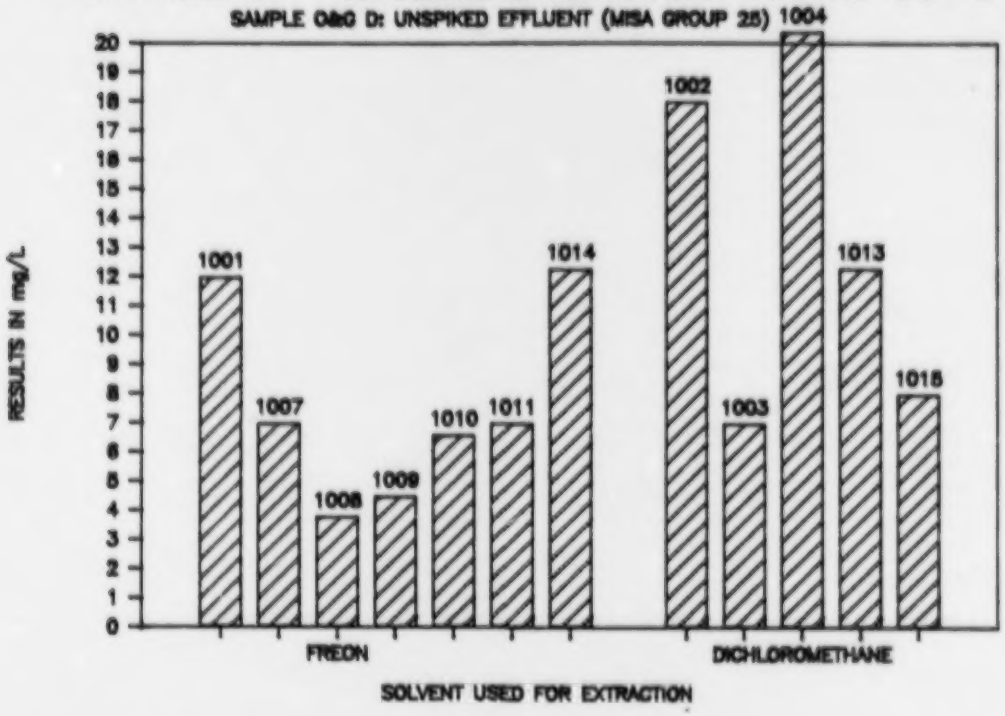
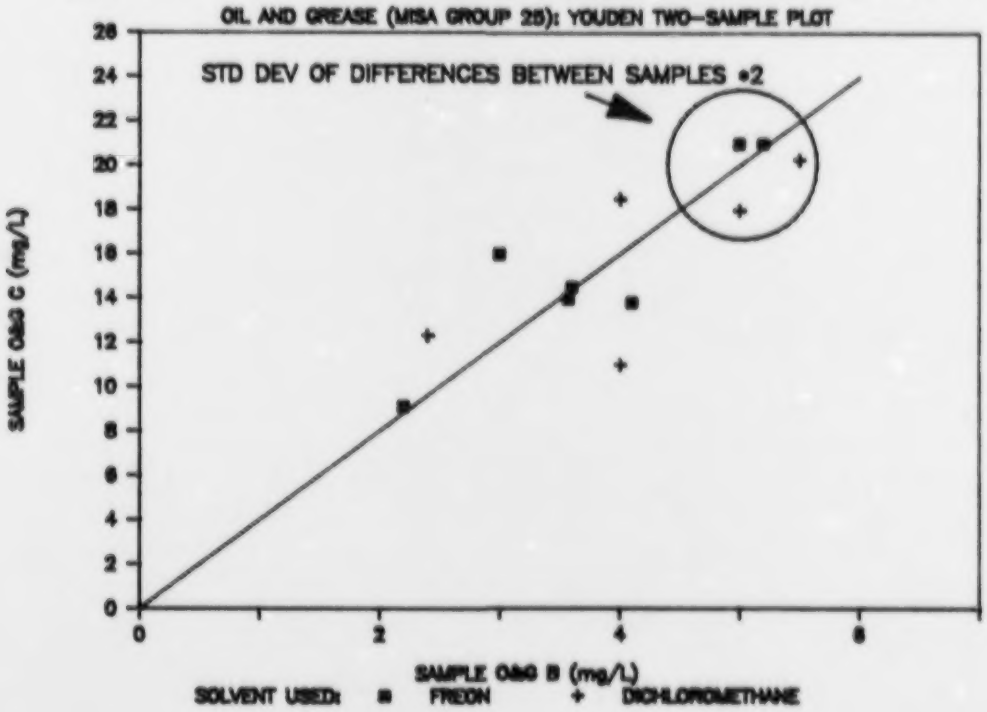


FIGURE 10 – INTERLABORATORY STUDY 89–1


OIL AND GREASE (MISA GROUP 2B): YOUDEN TWO–SAMPLE PLOT
STD DEV OF DIFFERENCES BETWEEN SAMPLES •2
SAMPLE O&G C (mg/L)
SAMPLE O&G B (mg/L)
SOLVENT USED: ■ FREON + DICHLOROMETHANE

7 APPENDIX 2 - LIST OF PARTICIPANTS AND CORRESPONDENCE

LIST OF PARTICIPANTS

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Mann Testing Laboratories Ltd.
5550 McAdam Rd.
Mississauga, Ontario
L4Z 1P1
(416) 890-2555

Contact: Tim Munshaw

Ortech International
2395 Speakman Dr.
Mississauga, Ontario
L5K 1B3
(416) 822-4111

Contact: Jack Brady

Zenon Environmental Inc.
845 Harrington Court
Burlington, Ontario
L7N 3P3
(416) 639-6320

Contact: Glenys Foster

Enviroclean
Environmental Laboratory Services
921 Leathorne St.
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N5Z 3M7
(519) 686-7558

Contact: William M. Neaves

Ontario Ministry of the Environment
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125 Resources Rd.
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M9W 5L1
(416) 235-5760

Contact: Yvonne Jones

Barringer Laboratories
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Contact: Alan Lipski

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85 Midpark Rd.
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Contact: Deborah Boersma

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P.O. Box 100
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Contact: Doug DeCoppel

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143 Dennis St.
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Contact: Karen Nicol

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Contact: Dr. Peter Child

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Contact: Paul Epstein

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3650 Wesbrook Mall
Vancouver, B.C.
V6S 2L2
(604) 224-4331

Contact: Dr. John Leach

MOE INTERLABORATORY VARIABILITY STUDY NOTIFICATION FOR THE ANALYSIS OF TRACE ORGANIC COMPOUNDS AND SOLVENT EXTRACTABLES STUDY NO. 89-1

INTRODUCTION

Private laboratories receiving this notification are invited by the Ontario Ministry of the Environment to participate in an interlaboratory variability study of spiked reagent water conducted using MISA analysis protocols. Laboratories interested in participating in this program, scheduled for the week of January 30, 1989, should contact Sylvia Cussion at (416) 235-5842 or Catherine Doehler (416) 235-6055 of the Ministry of the Environment to confirm participation no later than January 25, 1989. All participants should follow-up telephone acceptance with written confirmation (FAX - (416) 235-5744) by January 25, 1989.

BACKGROUND

This study is being conducted to assist laboratories in assessing their analytical performance. All procedures should follow those principles and protocols outlined in the MISA regulations (Ontario Reg. 358/88). Sample sets will include three samples per scan: a blank, a low spike (approx. 5 times the MDL) and a high spike (approx. 20 times the MDL). Sample sets for Oil & Grease (MISA Group 25 - Solvent Extractables) will include 4 samples: a blank, low spike, high spike, and a "natural" matrix sample.

NOTE: Any laboratory that does not have a copy of the MISA general regulations should contact Catherine Doehler for additional information.

The following scans are to be included in this round robin:

Volatiles (to be analyzed by GC/MS and/or GC/ECD/FID)
MISA Groups 16 & 17

Time Limit: 7 days storage

1,1,2,2-Tetrachloroethane
1,1,2-Trichloroethane
1,1-Dichloroethane
1,1-Dichloroethylene
1,2-Dichlorobenzene
1,2-Dichloroethane
1,2-Dichloropropane
1,3-Dichlorobenzene
1,4-Dichlorobenzene
Bromoform
Bromomethane
Carbon tetrachloride
Chlorobenzene
Chloroform
Chloromethane
cis-1,3-Dichloropropylene
Dibromochloromethane
Ethylene dibromide
Dichloromethane (Methylene Chloride)
Tetrachloroethylene
trans 1,2-Dichloroethylene
trans 1,3-Dichloropropylene
Trichloroethylene
Trichlorofluoromethane
Vinyl chloride (Chloroethylene)

Benzene
Ethylbenzene
Toluene
o-Xylene
m-Xylene
p-Xylene

Solvent Extractables (liquid/liquid extraction)
MISA Group 25

Time Limit: 7 days storage

Oil & Grease

SCHEDULE

During the week of January 30, 1989 participating laboratories will receive a total of seven (7) samples for analysis. Three (3) samples will be for the analysis of Volatiles and four (4) will be for the analysis of Solvent Extractables. All samples will be spiked reagent water samples, with the exception of Solvent Extractables, which will include a fourth unspiked sample.

Participating laboratories are expected to analyze the samples within the time limits specified in Schedule 2 of the general MISA regulations (Ontario Reg. 358/88). Blank reporting forms will be provided with the samples. Results for all analyses are to be reported within thirty (30) days of receipt of the samples to Sylvia Cussion/Catherine Doehler at the following address:

Ministry of the Environment
Laboratory Services Branch
Laboratory Computer Systems - QA/QC Section
125 Resources Rd., P.O. Box 213
Rexdale, Ontario
M9W 5L1

SUMMARY OF RESULTS

All participating laboratories will be assigned a unique identification code. All laboratories will receive a complete set of the results, including a ranking for each laboratory where they will be identified only by their identification code. Recommendations made by the MOE will also be provided to the individual labs. Results will remain confidential and will only be released with the written permission of the individual participants.

It is the intent of this round robin (along with others) to assess the interlaboratory variability and detection capability for a broad range of organics and inorganics.

Ontario Ministry of the Environment
Laboratory Services Branch
LCS-QA/QC Section
125 Resources Rd.
Rexdale, Ontario
M9W 5L1
(416) 235-5842 or 235-6055
FAX (416) 235-5744

January 31, 1989.

TO: PARTICIPANTS OF MOE ROUND ROBIN 89-1

Please find enclosed four (4) 1000 mL amber bottles and three (3) 40 mL clear bottles. The samples are labelled as follows:

1000 mL Amber Bottles	40 mL Clear Bottles
O&G A	VOL A
O&G B	VOL B
O&G C	VOL C
O&G D	

If you are missing any of the above items, please contact us at the above phone number immediately.

Your participation in MOE Round Robin 89-1 is greatly appreciated by the Laboratory Services Branch of the Ministry of the Environment.

As was stated in the notification distributed January 18, 1989, samples should be analyzed using the principles and protocols outlined in the MISA general regulation (Ontario Reg. 358/88). Store all samples in a refrigerator at 4 degrees Celcius until ready for analysis. Time limits for storage were indicated in the advance notification. Results are to be reported within thirty (30) days of reception of the samples. Report forms are included with the samples. Please identify all sample results with your lab identification number and the sample numbers described above. Please contact us if there are any problems or questions re the round robin.

Your lab identification number is:

Sincerely,

Sylvia Cussion
Lab Quality Audit Scientist
(416) 235-5842

Catherine Doehler
MISA Audit Scientist
(416) 235-6055

Laboratory Services Branch
LCS-QA/QC Section
125 Resources Rd.
Rexdale, Ontario
M9W 5L1
(416) 235-5842

June 14, 1989

TO: PARTICIPANTS OF MOE LINTERLAB VARIABILITY ROUND ROBIN 89-1

Thank you for your participation in the Interlaboratory Variability Study 89-1 conducted in January 1989. Your efforts are greatly appreciated by the Ministry.

Attached are all the results reported to me as of the end of March 1989. Not all the original participants were able to report results. Listed for each sample and parameter are the expected values, the results reported by each participant, the mean, maximum result, minimum result, and the standard deviation. For labs that reported a result for a particular parameter in Groups 16 & 17 for Samples B and C using a < or ND (not detected) designation, the value was entered as 0. In group 25, all < values were entered 1/2(<) for calculation purposes. All participants are designated by their identification code only.

A formal report is presently being written, but I regret that it will not be completed until July 1989. All participants will receive a copy as soon as it becomes available.

Please contact me if you have any further questions.

Sincerely,

Sylvia Cussion
Laboratory Quality Audit Scientist

Attachment